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Ceramic Powders Characterization: Results of an International Laboratory Study

S. G. Malghan and S. M. Hsu, Editors

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²Some elements at Boulder, CO 80303.

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S. G. Malghan and S. M. Hsu, Editors

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FORWARD

Reproducible manufacturing of high quality advanced ceramics components requires the use of powders having high purity, and consistent physical and chemical properties. The development and use of technically sound powder characterization methods is one of the ways to accomplish reproducibility in manufacturing. In addition, rapid commercialization of the advanced ceramics can be accomplished by reaching a consensus on the measurement methods on an international-scale since the raw materials and their processing to manufacture ceramic components is carried out in different countries. Instead of carrying out the required research in individual countries, the United States, Germany and Sweden joined hands in 1979 under the auspices of the International Energy Agency to initiate a collaborative research program on advanced ceramics for high performance engines and other energy conservation applications.

In 1984, under the far-sighted leadership of Messers Al Chesnes, and Robert Schulz, Office of Transportation Systems in DoE, Mr. Ed Lenoë of the U.S. Army Materials Command in Watertown, MA, an international cooperative study on advanced ceramics was launched. With the inputs of the U.S. industries, an agreement was formulated under the auspices of the International Energy Agency, IEA, and the countries involved were the United States, Germany, and Sweden.

The agreement is called "Implementing agreement for a program of research and development of high temperature materials for automotive engines". The U.S. Department of Energy is the Operating Agent for the agreement, with the Kernforschungsanlage Jülich GmbH in the Federal Republic of Germany, and the Swedish National Board for Technical Development as the counterparts for the agreement. The initial technical management was carried out by the U.S. Army Materials Command under contract to DoE. This was changed to the Oak Ridge National Laboratory in 1987.

The research in this report was conducted under the IEA Annex II Subtask 2 agreement. The objective of this task was to study the various characterization methods on ceramic powders. The study was coordinated by the National Institute of Standards and Technology in cooperation with U.S. Department of Energy. This report represents the combined efforts of many people in 25 organizations who participated in the ceramic powders interlaboratory comparison project. It also represents a landmark for international cooperation in the fast growing technology. We would like to express our sincere thanks to all who participated in the project, especially the national technical leaders: Dr. Alan Dragoo of the United States, Prof. Hausner of Germany, and Dr. Robert Pompe of Sweden.

To the readers of this report, this document is the first of its kind, and is the most comprehensive compilation of fine ceramic powder properties to date. In the future, we plan to prepare reports on the subsequent Subtasks 6 and 8.

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EXECUTIVE SUMMARY

The application of advanced ceramics in engines depends on developing reliable and cost-effective ceramics that will withstand harsh engine environments. Improvement in the characterization of ceramic starting powders is a critical factor in the control of reliability. Ceramic powders used by manufacturers of components are increasingly obtained from producers throughout the world. Consequently, the need for internationally agreed upon measurement procedures is becoming increasingly critical.

This report contains the results of an international interlaboratory comparison program ("round-robin") of powder characterization under the auspices of International Energy Agency (IEA). The results include the contributions of 25 industrial, university and governmental laboratories in the Federal Republic of Germany, Sweden and the United States. These laboratories surveyed a variety of methods for the characterization of physical properties and chemical characteristics of ceramic starting powders. For the first time, data from various measurement methods are available for critical comparison. Results of the same method from different laboratories are also provided. This represents an important milestone towards international consensus on powder measurement methodology in advanced ceramics.

The selected fine powders of silicon nitride, silicon, silicon carbide, and zirconia were representative of different methods of production and various types and stages of ceramic component manufacture. Careful preparation of the samples of the selected powders was carried out by the National Institute of Standards and Technology (NIST) to assure sample-to-sample homogeneity. Measurements were carried out by each laboratory according to its customary procedures. All results and descriptions of methods were reported to NIST. Data compilation and statistical analysis were performed by NIST personnel. The three technical leaders then performed analyses and drew conclusions. These conclusions were sent to individual participants for comments.

This report presents the final results and summarizes the data obtained and the methods studied under the auspices of IEA/Annex II, Subtask 2, Powder Characterization. Recommendations for future studies are drawn from results of the present round-robin. The data obtained in Annex II will no doubt serve as the basis for designing and conducting future studies as well as for the development of standard procedures needed in the international arena of advanced ceramic materials.

Technical Program Description

Samples of the following five powders: silicon nitride (reference), silicon nitride (test), silicon, silicon carbide and zirconia, were prepared and sent to the participating laboratories. Each laboratory performed a core group of analyses using standard methods employed in their respective laboratories. A summary of properties and methods is presented below.

I. Physical Properties

Density

Bulk density (helium pycnometer)

Tap density

Specific surface area

Single point BET

Multi-point BET

Particle size and size distributions

Gravitational sedimentation

Centrifugal sedimentation

Light scattering

Fisher sub-sieve

Photon correlation spectroscopy

Electrical sensing zone

Particle Morphology

Scanning Electron Microscopy

Transmission Electron Microscopy

Phase Composition

X-ray diffraction

II. Chemical Properties

Major Methods

X-ray fluorescence (XRF)

Atomic absorption (AA)

Inductively coupled plasma (ICP)

Direct current plasma (DCP)

Gravimetry

Major Non-Metallic Impurities

Total carbon in Si_3N_4 , SiC (Combustion)

Free carbon in SiC (Combustion)

Total carbon in ZrO_2 (Combustion)

Nitrogen in SiC , Si , Si_3N_4 (Combustion)

Oxygen in Si_3N_4 , SiC , Si (Fast Neutron Activation Analysis)

Sulfur in Si_3N_4 (Combustion)

Fluorine in Si_3N_4 , SiC (Ion Chromatography and
Electrochemical Method)

Chlorine in Si , SiC (Ion Chromatography)

Sulfur in ZrO_2 (Inductively Coupled Plasma)

(Combustion)

(Mass Spectrometry)

(Ion Chromatography)

Chlorine in ZrO_2

(Ion Chromatography,
Neutron Activation and
Argentometric Titration)

Major Metallic Impurities

Free Si in Si_3N_4

(Electrochemical Titration)
(Volumetric Analysis)

Al, Fe, Ca in Si_3N_4 , SiC, Si (Atomic Absorption)

(Inductively Coupled Plasma)
(Direct Current Plasma)
(X-ray Fluorescence)
(Mass Spectrometry)

Minor Impurities

S, F, Cl, Na, Mg, K, Ti, Cr, Mn

Ni, W, B in Si_3N_4 , Si

(Inductively Coupled Plasma)
(Atomic Absorption)
(Mass Spectrometry)
(Direct Current Plasma)

Results and data collected from the participants were analyzed statistically at NIST by Dr. R. Munro to identify outliers and the primary sources of variance. Binary comparisons of data and methods were also performed.

Conclusions

Average particle size, density, and surface area are in good agreement not only among different laboratories but also among the various methods used. This is somewhat contrary to the previous experience on ceramic powder interlaboratory comparison. The major difference this time is the extraordinary care taken in sample preparation and packaging. This resulted in a minimum sampling error.

Particle size distributions from six methods showed a wide range of variation. The cause of the variation had been determined to be mainly from the following three sources: sample pretreatment method and severity, different sensitivity ranges from various instruments, and different measurement principles.

The chemical analysis of major elements and minor trace impurities presented a higher than normal deviation. The source of error can be traced to: the lack of standard reference materials for calibration; different sample pretreatment methodology associated with different instruments; the choice of instruments and methods.

Specific conclusions are as follows:

1. Sample preparation methods influence the results of particle size measurements. Consequently for a set of methods to be compared, a universal prescription for sample preparation is required so that a stock slurry is obtained which satisfies (1) the volume requirement of the method with the largest volume requirement and (2) the concentration requirement of the method with the most concentrated suspension. Volumes and concentrations for other methods to be compared would be obtained by taking suitable aliquots and by making the appropriate dilutions.
2. A bias between specific surface areas determined by single point and multipoint BET measurements was apparent. However, accurate evaluation of this bias was obscured by the variability in the data. One source of variability was the use of different outgassing times and temperatures. Establishment of a common preparation procedure is recommended.
3. A limited amount of quantitative x-ray diffraction data for phase content determination was obtained. Several methods for analysis of the data were used by the laboratories. Concentration (in percent) of secondary phases typically varied by about 2%. Some of the methods used were outdated or contained measurement biases. A wider base of test data for methods based on the best measurement assumptions and protocols is needed. The use of silicon nitride as the initial test powder is suggested.
4. Results showed low precision (relative standard deviation > 25 %) for the analysis by AAS, ICP and DCP of the major metallic impurities Al, Ca and Fe in Si_3N_4 and SiC. Better interlaboratory agreement (i.e., reproducibility) and intermethod consistency were obtained with acid digestion rather than with flux dissolution of Si_3N_4 and SiC. More problems were evident in the analysis of SiC than of Si_3N_4 . Within-laboratory uncertainty (i.e., repeatability) was improved by the use of an internal standard, such as Sc for ICP, and by the use of synthetic calibrating solutions.
5. Limited data on halide impurities in Si_3N_4 and SiC were obtained. Two sample preparation methods were used for extraction of halides: hot-water leaching and flux decomposition. Leaching methods were employed in a limited manner, mostly for F and Cl impurities. Both hot water and boiling water leaching were used. However, insufficient data were obtained for detailed analyses. Principal detection methods used were: ion chromatography, specific ion electrode, argentometric titration for Cl^- and potentiometric titration. Concentrations determined after leaching were somewhat less than those obtained after decomposition.
6. Oxygen in Si_3N_4 and SiC was determined by combustion/decomposition using either thermal conductivity or IR absorption detection. The results of most laboratories were in agreement with results from fast neutron activation analysis; however, some results for O in Si_3N_4 (both reference and test samples) were inexplicably high.

7. Results for total C in SiC showed low precision (relative standard deviation > 3 %) and a possible negative bias of about 2%. Carbon analysis by combustion/decomposition may be improved by the development of better calibration standards.
8. Kjeldahl analysis of N in Si₃N₄ and Coulometric titration of C in SiC were each tested by three laboratories. Both methods require decomposition of the sample followed by recovery of the element in an evolved gaseous compound and then by analytical determination. The overall precision was good in both cases, and the development of reference methods based on these two approaches appears to be promising. However, specific procedures need to be developed and tested in more laboratories.

Recommendations. Recommendations for further comparisons of powder characterization measurements are discussed at length in the report. The recommendations were developed by the task leaders in their analysis of the data, through further discussions between themselves and in consultation with participants. The recommendations may be summarized as follows:

1. Further interlaboratory testing is required to improve the analysis by AAS, ICP and DCP of Al, Ca and Fe in Si₃N₄ and SiC.
2. Further interlaboratory testing of an analysis procedure for halides is required to develop sufficient data for an accurate evaluation by leaching or decomposition analysis.
3. Develop and evaluate procedures for analysis of C and O in Si₃N₄ and SiC by combustion/decomposition.
4. Develop and evaluate a potential reference method for one of the following analyses:
 - a. Determination of N in Si₃N₄ by the Kjeldahl method;
 - b. Determination of C in SiC by Coulometric titration.
5. Develop and evaluate a method for analysis of free-carbon in SiC, similar to DIN 51075 (in FRG) which was intended for coarse-grain SiC.
6. Test one or more methods for the analysis of discrete impurities.
7. Develop and test a methodology for sample preparation which enables two or more methods to be compared. SEM analysis of deposited particles is suggested as a method for evaluation of agglomeration conditions in the slurry.
8. Develop a sample preparation procedure for BET measurements and evaluate the bias between single point and multipoint methods.
9. Compare thermal weight loss measurements for a selected powder.

10. Evaluate one or more methods for spectroscopic analysis of powder surface chemistry.
11. Evaluate and compare methods based on current laboratory practice for determination of surface charge related properties using such methods as electrokinetic measurements and acoustophoresis.
12. Devise and evaluate a procedure for quantitative phase determination by x-ray diffraction.

Subsequent to the completion of the Subtask 2 work, the participants voted to continue the powder characterization task under Subtask 6.

The contributions from all participants in the round robin are gratefully acknowledged.

ACKNOWLEDGMENTS

The research described in this report and conducted under the terms of the International Energy Agency (IEA) Annex II agreement entitled, "Implementing Agreement for a Programme of Research and Development of High Temperature Materials for Automotive Engines," was performed by participating laboratories in the United States, the Federal Republic of Germany, and Sweden. The research in this report is identified within the Agreement as Subtask 2, Powder Characterization.

The organization in each country responsible for the research was the Department of Energy in the United States, the Kernforschungsanlage Jülich GmbH (KFA-PLR) in the Federal Republic of Germany, and the Swedish National Board for Technical Development (STU) in Sweden. The person in each country responsible for organization and overseeing the research was A. L. Dragoo at National Institute of Standards and Technology (NIST) in the United States; H. Hausner, Technical University of Berlin, Federal Republic of Germany; and R. Pompe, Swedish Ceramic Institute, Sweden. Messrs. Dragoo, Hausner, and Pompe also were responsible for conducting the technical analysis of the data. A. L. Dragoo provided extensive preparation and editorial work on this report, and brought together the individual contributions. S. G. Malghan provided descriptions of many of the methods and made a major contribution in the preparation of the report. L.-S. H. Lum and R. Munro provided invaluable help in statistical analysis of the data. L.-S. H. Lum also prepared the final data tables. M. Robinson was responsible for typing the report. S. M. Hsu, Chairman, of NIST coordinated all of the research activities in Subtask 2.

V. J. Tennery, Oak Ridge National Laboratory, had overall responsibility for this IEA Agreement research in the United States. We want to acknowledge the support of D. R. Johnson, Project Manager, Oak Ridge National Laboratory, under Ceramic Technology Program this work was conducted. Messrs. R. B. Schulz and A. Chesness provided an overall leadership and guidance, and a significant financial support to NIST for this work. The Department of Energy, through the Heat Engine Propulsion Division, Office of Transportation Systems, provided partial financial support (the Ceramic Technology for Advanced Heat Engines Project of the Advanced Materials Development Program, under Contract DE-AC05-84OR21400, with Martin Marietta Energy Systems, Inc.). The researchers in many laboratories in the three countries who contributed their knowledge and skills to this research which focused on the evolution of standardized ceramic powder characterization measurements are too numerous to name here, but their participation was critical to this international cooperative effort.

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1. INTRODUCTION

S. G. Malghan and A. L. Dragoo
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1.1. BACKGROUND

Purpose of IEA/Annex II

The IEA/Annex II program was directed toward generic research in high-temperature structural ceramics for advanced engines and other energy conservation applications. The major objective was the evolution of internationally accepted standardized testing and characterization methods for structural ceramics and for starting powders used in the fabrication of structural ceramics.

Program

The Annex II program set up three major experimental tasks to achieve its objective. These tasks were:

- characterization of ceramic powders (Subtask 2)
- characterization of structural ceramics (Subtask 3)
- measurement of fracture strength (Subtask 4)

Annex II was developed on the basis of work completed in Annex I which emphasized ceramic component development for gas turbines. Industry and government laboratories worked closely in both activities to define the technical scope of work, to carry out the measurements and to discuss and to evaluate the results. Industrial participants in each country cost-shared a portion of the research.

During its formative stage, Subtask 2 was under the chairmanship of Dr. J. W. McCauley, U.S. Army Materials Technology Laboratory. During this stage, NIST provided technical support through the preparation and distribution of samples. During the second stage of Subtask 2 which required the compilation, distribution of results to participants and data analysis, Dr. S. M. Hsu, NIST, served as the chairman.

Importance of Advanced Ceramics for Heat Engines

Ceramics have been successfully employed in engines on a demonstration basis. The successful manufacture and use of ceramics in advanced engines depends on the development of reliable materials that will withstand high, rapidly varying stress loads. Improvements in the characterization of ceramic starting powders is a critical factor in achieving reliable ceramic materials for engine applications.

1.2. PURPOSE OF SUBTASK 2

Objectives

The overall objective of the powder characterization effort was the enhancement of the quality, reproducibility and reliability of powder characterization measurements and, consequently, of ceramic powders.¹ Through a program of interlaboratory comparison of measurements, the participants in Subtask 2 sought the following:

- survey methods currently in use for characterization of ceramic starting powders,
- establish a base of data and experience which would identify unique sets of properties and methods useful for powder evaluation and comparison,
- determine the extent of agreement between laboratories,
- identify areas for further measurement research, and
- recommend useful standards which would aid international commerce.

Participants were encouraged to report results of measurements which they believed represented the "true" values of the material characteristics.

Importance of Powder Characterization

Physical and chemical characteristics of powders strongly influence their behavior during ceramic processing and, thus, impact ultimately upon the microstructure and performance of ceramic components produced from them. The ceramic process cannot be adequately controlled for large scale production of high-quality technological components without detailed information on the powder characteristics. Moreover, as identified by the NIST study conducted by Charles River Associates² (CRA), the key issues in the commercialization of ceramic materials for advanced technological applications are production cost, reliability in service and reproducibility in manufacture. For the case of ceramic heat engines, CRA saw reliability as a major technological barrier, and improved characterization of ceramic powders as providing "important leverage in overcoming the reliability and reproducibility problems." Improved characterization of ceramic powders requires not only improved measurements but also improved understanding of how powder characteristics are related to properties and performance of resulting ceramic materials.

In view of the many applications of structural ceramics, as heat engine components as well as in other areas of use, the analysis of powder characteristics includes particle size distribution, particle morphology, bulk elemental composition, surface chemistry, crystalline/noncrystalline phases, surface area, density, porosity, and grain or primary particle size. Different applications emphasize different combinations of these characteristics.

Importance to Industry and Commerce

Powders used in the fabrication of advanced structural ceramic components are manufactured in a number of the major industrial countries of the world. A manufacturer of ceramic components may obtain powders from a number of these sources. However, few standard methods and certified reference materials are available for the evaluation of these powders in the international marketplace. Moreover, a set of data obtained from characterization of a large number of homogeneous samples by several laboratories was not available, prior to this study, to assist in the development of standard methods and reference materials.

A key part of Subtask 2 was the participation of companies from the three countries. Powders (See Section 2.1.1, below) were obtained from different sources. Current characterization instruments and procedures were compared. Finally, a forum was provided in which measurement methods and problems were discussed.

1.3. TECHNICAL BACKGROUND

Issues in powder characterization are what to measure, how to measure and how to ensure quality in analytical measurements. "What to measure" involves understanding the relationships between powder characteristics and ceramic microstructures. "How to measure" requires the development of measurement methods and the determination of repeatability and reproducibility. A thorough analysis of repeatability and reproducibility requires breaking a method down into a series of procedural steps and determining the influence of each step on the precision and, if possible, on the accuracy of the measurement. Standard (or certified) reference materials are required to assure the quality of the measurements and comparability of measurements between different laboratories and techniques or equipment.

A foremost requirement to achieve these objectives is the selection and preparation of powder samples. Repeatability and reproducibility of ceramic powder characteristics are assured only when the powder samples are prepared according to well-established statistical procedures.

Powders

The powders were selected on the basis of extensive discussion and consensus among the participants. Several factors were considered in these discussions: (1) production in one of the participating countries, (2) use by one or more companies in the manufacture of structural ceramics, and (3) use in the fabrication of samples for Subtasks 3 and 4. The powders selected for the program, along with the abbreviations used for them in this report were:

Silicon nitride, LC-10^(a), H.C. Starck (SNR)
Silicon nitride, SNE-10, Ube Industries (SNT)
Silicon, IV-D, Kemanord (Si)
Silicon carbide (< 5 μ m), H.C. Starck (SiC)
Yttria-zirconia, *ToyoSoda (YSZ)

The LC-10 silicon nitride powder was selected by the participants to serve as an internal reference material for the purposes of the round-robin.

*Since completion of this work ToyoSoda has changed company trade name to TOSOH

The powder lots were obtained by Oak Ridge National Laboratory and delivered to NIST for sampling, distribution and archiving.

Sample Preparation: Summary of Procedure for Subdivision of Powders.

Sample preparation required:

- (1) the division of a bulk lot of powder into representative samples,
- (2) the avoidance of contamination, and
- (3) packaging in a manner which would assure long-term sample integrity.

Division of the powder by means of spinning riffles was used to minimize sample-to-sample variance. Contamination was avoided by processing one powder at a time and by operating the riffles in an isolated laboratory which was serviced by a filtered air supply. Powders were stored under an inert gas. Sample vials were hermetically packaged with a dry argon ambient in the free space of the vials and surrounding envelope.

Powder samples were prepared from 25 kg bulk lots of powder, with the exception of the LC-10 silicon nitride which was received in a 100 kg lot. Sample preparation consisted of cone blending of the total lot, followed by manual division by small increments into several plastic lined metal containers to obtain units containing about 2 to 3 kg each, depending upon the packing density of the powder. Thus, a 25 kg lot was split into 8 or 12 smaller lots. Due to the fluffiness of the LC-10 powder, it was split into 48 smaller lots. These units were vacuum dried and the containers were backfilled with dry argon.

Two subsequent divisions were carried out using spinning riffles, such as the one shown in Figure 1. A third riffing was performed using a smaller

^(a) Certain commercial equipment, vendors, instruments, or materials are identified in this report in order to adequately describe experimental requirements. In no case does such identification imply recommendation or endorsement by the International Energy Agency, nor by any of the participants in IEA/Annex II, nor does it imply that the vendor material or equipment identified is necessarily the best available.

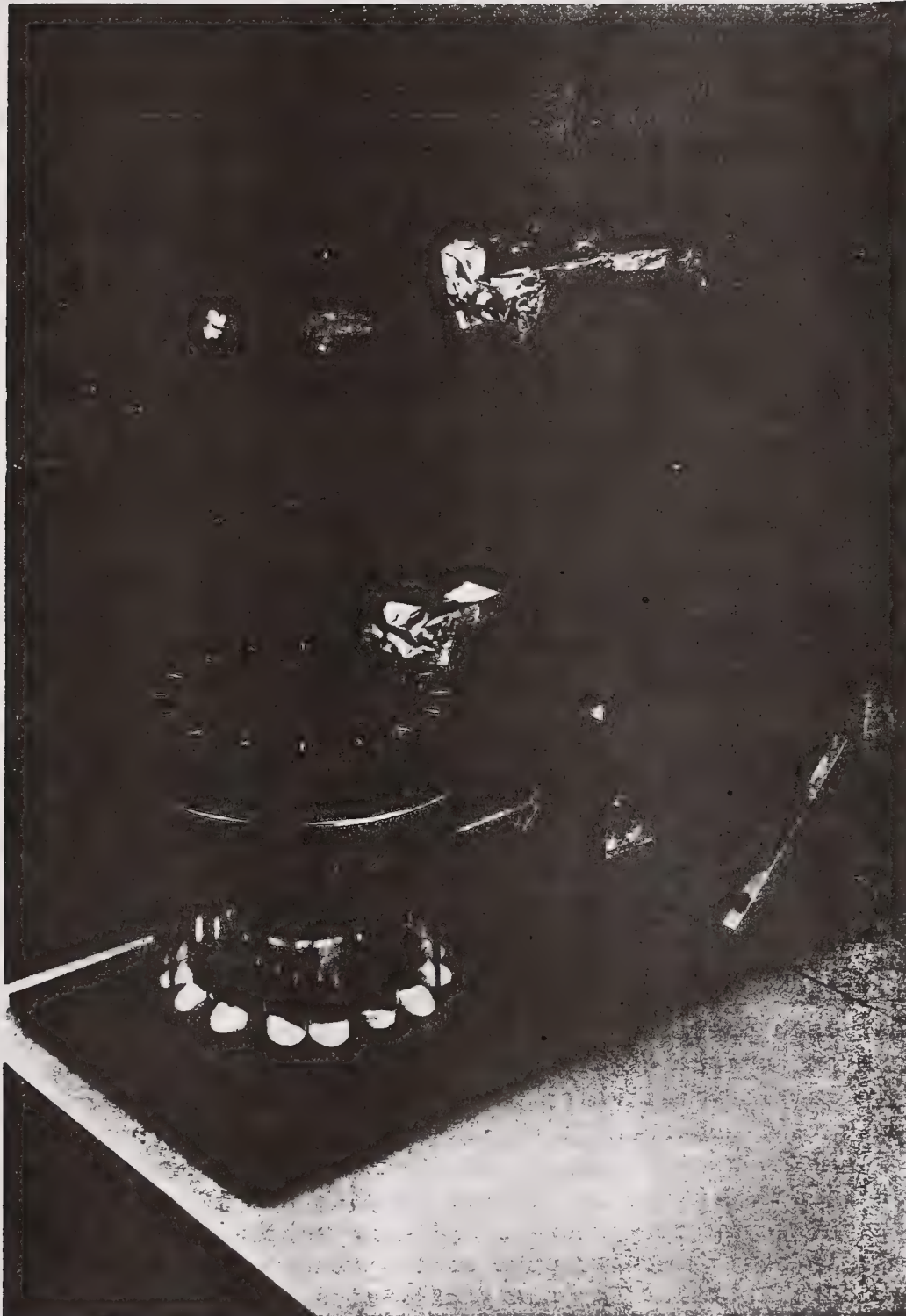


Figure 1. Laboratory spinning riffler

riffler. Samples were vacuum dried and backfilled with dry argon after each operation. The final riffling was carried out entirely in an ambient of dry argon, and the samples were collected directly in sample vials which were closed with plastic caps; see the inner sample vial shown in Figure 2. The samples which were obtained from the final operation were about 4 g in weight for the silicon nitride, silicon carbide and silicon samples and 12 g in weight for the yttria-zirconia samples. These samples were hermetically sealed immediately in foil laminate pouches to preserve an atmosphere of dry argon around the vials. For the yttria-zirconia powder and the two silicon nitride powders, sample vials were subsequently inserted individually into an outer glass envelope which was flame-sealed. A schematic of a flame-sealed sample is illustrated in Figure 2. A major equipment failure with the flame-sealer prevented the use of this encapsulation method for the silicon and silicon carbide samples.

Sample-to-Sample Homogeneity

Sample-to-sample homogeneity was maintained (1) by the use of spinning riffles, (2) by probability sampling at all stages and (3) by blending randomly selected pairs of samples prior to riffling. Verification of homogeneity was carried out by testing randomly selected samples and by maintaining traceability of samples. Traceability required maintaining a lineage record for each sample so that sample numbers associated with anomalous measurement values could be compared.

1.4. MEASUREMENTS

The major intent of this round-robin was the survey of a variety of methods which provide characterization of the powder chemistry (i.e., elemental constituents and phases), microstructure, particle size, other physical properties and processing defects. For the purpose of the most complete statistical analysis of the data it was desirable for each participant to contribute sufficient characterization of as many selected properties as possible. However, the analyses selected and the extent of the effort of each participant depended on their interest, experience and resources. The degree to which a single method was replicated by a single laboratory or used by several laboratories varied widely in this study. Although an attempt was made to have at least four laboratories using the same method, this was not always realized in the final results. In some cases in the following report, results contributed by one or two laboratories are considered and compared with results obtained by other methods because the precision of the measurement, possible accuracy of the method, or because degree of agreement with another method suggests that a more extensive study may be warranted in the future.

Each laboratory was instructed to follow their customary procedure for a method and to report what they considered to be the "true" value given by a measurement. Laboratories were requested to provide the individual results of replicate measurements and to provide documentation for the measurement procedure followed.

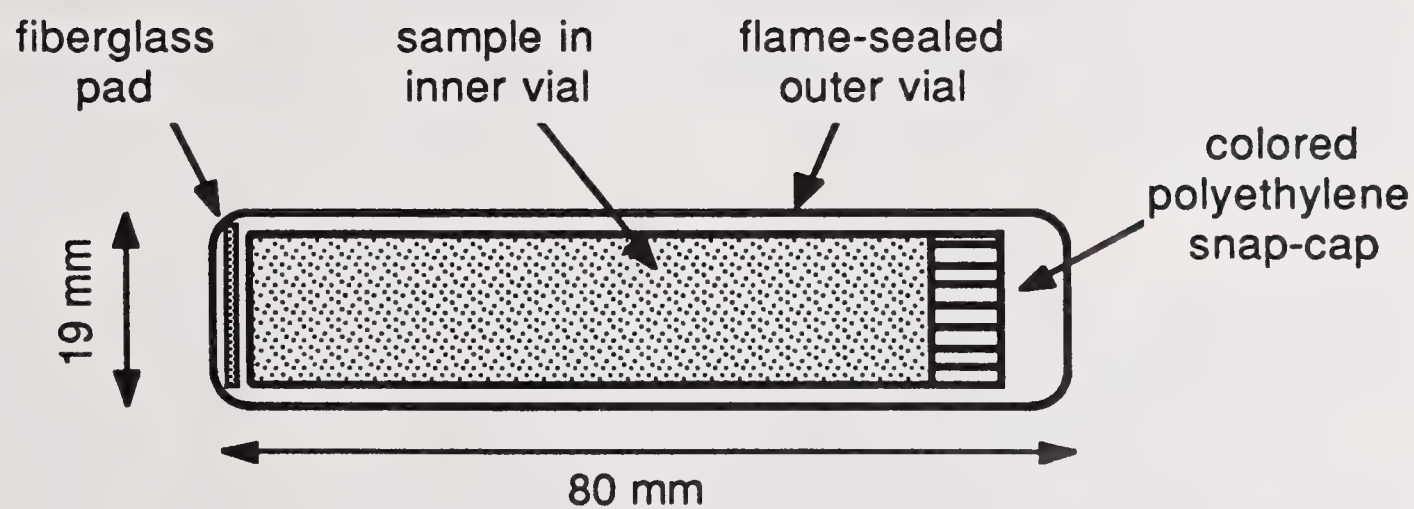


Figure 2. Illustration of flame-sealed sample.

Structure of the Report

The report is divided into several chapters, and each chapter is devoted to a particular property of the powder. In each chapter, the technical material is organized as follows:

- Methods used for making measurements.
- Statistical data in the form of tables and plots.
- Tabulated data from all participants.

Reporting of Results

For the purposes of comparison of the large volume of data in this report, as well as to reduce a large volume data to a tractable quantity for publication, it was necessary in most instances to report the results of a measurement by one or two numbers. In cases where an averaged bulk property is obtained by a measurement, no information is lost. However, for measurements of particle size distributions, in particular, this meant a representation of the distribution by a mean value, usually the median (D_{50}), and a measure of the spread, i.e., the difference of the size at 90% finer and 10% finer divided by D_{50} , or

$$(D_{90} - D_{10})/D_{50}$$

Although such a representation was in most cases an oversimplification and meant that the full details of the distribution were not reported here, the use of the central tendency and the spread may serve for an adequate comparison of measurements by different laboratories.

In addition, each laboratory was encouraged to run the H. C. Starck LC-10 silicon nitride as an internal reference material. Further comparison between laboratories can be made by first normalizing their results to their respective results obtained for the reference silicon nitride powder. A large body of data on this powder was collected by the participants.

Since most laboratories also provided selected information about the experimental procedures which they used, a limited number of analyses of the effects of analyte processing on measurements was possible. These analyses are presented as part of the technical analyses in each chapter.

1.5. STATISTICAL ANALYSIS⁴

There were 39 different properties that could be reported for each of the approximately 700 IEA samples. Each participating laboratory performed a selected subset of these tests, consistent with the measurements normally performed by that laboratory. As a result, laboratories performing physical measurements often reported particle size results, while laboratories conducting elemental analyses usually reported results for a selected set of chemical elements.

The data obtained in this study, therefore, are useful for identifying overall, or pooled, sources of variance, but not individual contributions from distinct sources such as sampling technique, specimen preparation, apparatus, and operator performance. That level of detail will be addressed in the future when specific measurement procedures are studied. For the present purposes, it is adequate to consider only effective measures of repeatability and reproducibility.

The variability in the property values determined by a given laboratory using a specified technique can be estimated by computing the standard deviation of the replicated measurements, i.e., the estimate of repeatability is based on the within-laboratory variance. An estimate of the reproducibility of a measurement can be made by computing the standard deviation of the means obtained from different laboratories using what is nominally the same technique; i.e., the estimate of reproducibility is based on the between-laboratory variance. It should be emphasized that this estimate in particular neglects differences in specimen preparation and detailed measurement procedures. When two or more methods are used to determine a property value, an overall mean can be computed for each method; then, an estimate of the robustness of a property measurement can be made by computing the standard deviation of the overall means obtained from each of the different methods.

Throughout this report, the terms "average value" (A) and "standard deviation" (S) for a set of N observations, $(x_1, x_2, \dots, x_N)$, are defined as follows:

$$A = (x_1 + x_2 + \dots + x_N)/N$$

$$S^2 = [(x_1 - A)^2 + (x_2 - A)^2 + \dots + (x_N - A)^2]/(N - 1).$$

These terms are used particularly in all of the tables of summary statistics in the following chapters. Each summary table has an identifying header and six columns of data. Each column has a column header which is self-explanatory, except perhaps for the first column. The first column, LAB #, identifies the sources of data for each row of the table. The source of the data for each row is a participating laboratory, except for the last row of the table which is separated from the primary rows of the table by a solid horizontal line. The entry for the LAB # in the last row of the table is always the word "Means". The source of data for this row is the column labeled "Average Value" in the primary rows of the table. Thus, the value of the standard deviation given in each primary row of the table is related to the repeatability of the measurement performed by one laboratory, while the value of the standard deviation in the row labeled "Means" is related to the reproducibility of the specified measurement across all of the laboratories.

Representative examples of the measures of precision, reproducibility, and robustness are presented here for both physical and chemical powder characterization measurements. Bulk density, tap density, mean particle size, and specific surface area are discussed as physical characterization measurements. For chemical characterizations, three illustrative cases of quantitative elemental analysis are discussed: measurements of a major constituent (nitrogen in silicon nitride), a nonmetallic impurity (oxygen in all of the powders except zirconia), and a metallic impurity (iron in all of

the powders).

Overall, the most persistent trend in the results is that the between-laboratory variance is greater than either the within-laboratory or between-methods variances. This trend bodes well for the effort to achieve standardization in powder characterization and indicates that the careful attention given to the primary sample preparation was successful. With the sampling error reduced, it appears that good precision can be attained within individual laboratories for each of the measured properties using a variety of measurement procedures. Further examination of the descriptive information supplied by the participants suggests that the quantitative differences among the values measured by different laboratories arose from the varying sample pretreatments, as well as from differences in instrumentation. These sources of variance can be reduced by standardized measurement procedures and will provide a focus for the next IEA round-robin program.

1.6. REFERENCES

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2. DENSITY OF POWDERS--METHODS AND DATA ANALYSIS

H. Hausner, Technische Universität Berlin; S. G. Malghan, NIST

2.1. METHODS

2.1.1. Bulk Density

Use and Significance

Bulk density of powders is a measure of the true (or skeletal) density plus closed internal porosity. This measurement is required for the application of several methods in particle size analysis.

Principle

The helium pycnometer is the most commonly used instrument for measuring the volume of bulk powders since helium will penetrate all of the open porosity. The volume of the powder is determined by the volume of gas displaced by the sample.

Instrument and Experimental Parameters

It is desirable to use as large a sample as possible to maximize the volume of gas displaced. Prior to the measurement, the sample is placed in a sealable sample holder and is degassed by heating the powder to 100 - 200°C under vacuum for a predetermined length of time. Following degassing, the sample is cooled in a vacuum desiccator. The cooled sample and sample holder are weighed and then transferred to the helium pycnometer. Volume measurements are carried out according to the procedure required for the particular instrument.

Calibration and Standards

Frequent calibration of the pycnometer is desirable to establish close experimental control of the instrument. This can be done using a steel ball which is provided as a reference for the instrument or by using a standard powder. The standard procedure, ASTM D854-83, "Standard Test Method for Specific Gravity of Soils," may be consulted for guidance in the determination of the specific gravity of ceramic powders.

Results and Discussion

Data are summarized in Section 2.3.

The bulk density measurements showed an excellent agreement, as can be seen in Table 2.1.1. This is certainly due to the fact that the same method, or even the same instrument type, was used and that in the latter case a well-specified procedure was followed.

Table 2.1.1. Range of Bulk Density Measurements (He-Pycnometer)

Material	Number of labs	Number of Determ.	Density (g/cm ³)		Calc. Data	Ref.
			Min.	Max.		
Si	2	57	2.30	2.35	2.33	1
SiC	2	54	3.08	3.19	3.21	2
SNR	2	34	3.12	3.18	3.18	3,4
SNT	2	51	3.13	3.20	3.18	3,4

Conclusions

The results of the bulk density measurements are in excellent agreement, but it has to be taken into consideration that only two laboratories using the same method reported results.

2.1.2. Tap Density

Use and Significance

The powder tap density gives an indication about the compaction behavior of the powder. The tap density of a dry powder is affected by the relative movement of individual particles to fill voids within the powder mass. This relative movement will be affected by the degree of agglomeration or dispersion of the particles. This property is also useful for comparing the flow properties of various powders in bins, chutes and conveyor belts.

Principle

The tap density is a relative measure of the fill density and is measured by dividing the weight of the powder by its volume after a specified number of taps at the bottom of the receptacle containing the powder.

Instrument and Experimental Parameters

A known weight of powder (e.g., 100.00±0.02 g) is poured into a graduated cylinder which is located in a tapping device for which the number of taps are preset. The measured tap density is strongly dependent on the manner in which the powder is loaded, number of taps, force imparted with each tap and wall effects of the graduated cylinder. The experimental conditions (e.g., the number of taps) vary significantly among different laboratories, rendering the comparison of results difficult.

Calibration and Standards

For guidance in conducting tap density measurements on ceramic powders, the following standard procedure may be consulted.

ASTM D3347-86. Flow rate and tap density of electrical grade magnesium oxide for use in sheated-type electric heating elements.

Results and Discussion

Data are summarized in Section 2.3. The results of five powders are compared in Table 2.1.2.

The values for the tap density show a large scatter which appears to be due largely to the number of taps used by different investigators.

Table 2.1.2. Range of Tap Density Measurements

Material	Number of labs	Number of Determ.	Density (g/cm ³)	
			Min.	Max.
Si	3	3	0.78	1.08
SiC	5	5	0.68	0.83
SNR	7	7	0.74	0.87
SNT	9	9	0.66	0.78
YSZ	2	2	1.49	1.51

Conclusion

Tap density values show a large scatter due most likely to different experimental procedures used by the participating laboratories.

2.2. DATA

2.2.1. Laboratories Making Density Measurements

Three types of density measurements were reported: bulk density, tap density and liquid density (Archimedes' method). The identification numbers of the laboratories making measurements are given in Table 2.2.1 for bulk and tap density.

Table 2.2.1. Laboratories Making Bulk and Tap Density Measurements

Powder Type	BULK Lab No.	TAP Lab. No.
Silicon	17, 23	6, 15, 18
Silicon Carbide	17, 23	6, 10, 15, 18, 21, 25
Silicon Nitride, R	17, 23	5, 6, 18, 21, 25
Silicon Nitride, T	17, 23	14, 15, 18, 21, 25
Zirconia	17, 18	6, 14, 18

For method codes, see Table 2.3.1.

2.2.2. Bulk, Tap and Liquid Density of Silicon Nitride Reference Powder

Table 2.2.2.1 Bulk Density by He Pycnometer

Material: Silicon Nitride, Reference Powder
Property: BULK
Method: 4

Lab #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
17	25	3.1200	3.1800	3.1386	0.0193
23	7	3.1310	3.1570	3.1421	0.0100
Means	2	3.1386	3.1421	3.1404	0.0025

Table 2.2.2.2 Tap Density

Material: Silicon Nitride, Reference Powder (Figure 3)
Property: TAP
Method: 3

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
5	2	0.74	0.78	0.76	0.028
6	1	0.87	0.87	0.87	0.000
15	2	0.77	0.77	0.77	0.000
17	3	1.46	1.48	1.47	0.010
18	1	0.74	0.74	0.74	0.000
21	1	0.71	0.71	0.71	0.000
25	1	0.79	0.79	0.79	0.000
Means	7	0.71	1.47	0.87	0.268

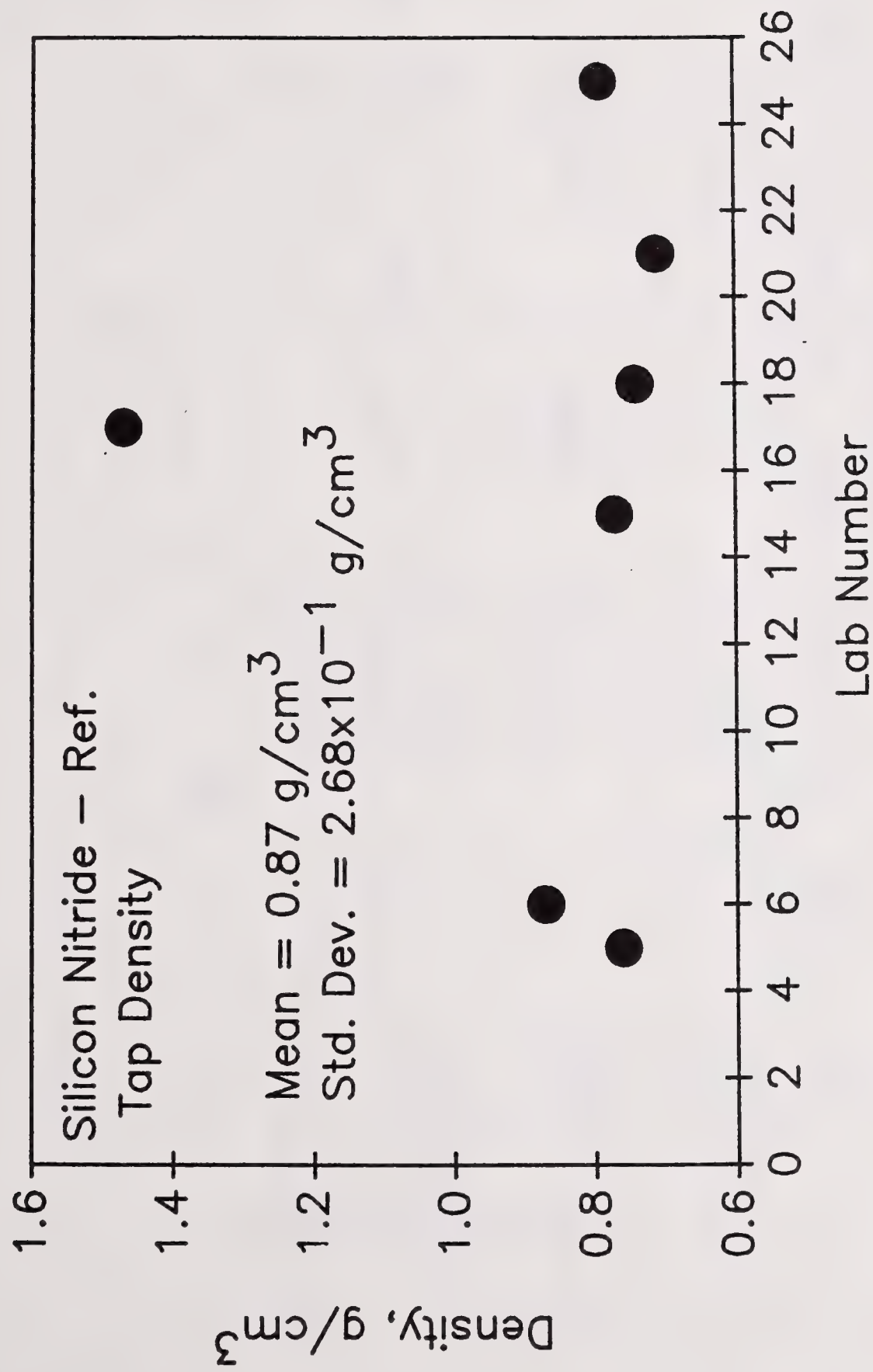


Figure 3 Tap density of silicon nitride (ref.) powder reported by participating labs. Lab numbers are given on the horizontal axis.

Table 2.2.2.3 Liquid Density in Hydrocarbon, Archimedes Method

Material: Silicon Nitride, Reference Powder
 Property: LIQUID
 Method: 6

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
21	1	0.56	0.56	0.56	0.00
25	1	0.48	0.48	0.48	0.00
Means	2	0.48	0.56	0.52	0.05

2.2.3. Bulk, Tap and Liquid Density of Silicon Nitride Test Powder

Table 2.2.3.1 Bulk Density by He Pycnometer

Material: Silicon Nitride, Test Powder
 Property: BULK
 Method: 4

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
17	43	3.1300	3.2000	3.1619	0.0148
23	7	3.1360	3.1700	3.1497	0.0130
Means	2	3.1497	3.1619	3.1558	0.0086

Table 2.2.3.2 Tap Density

Material: Silicon Nitride, Test Powder (Figure 4)
 Property: TAP
 Method: 3

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
6	1	0.76	0.76	0.76	0.00
14	2	0.65	0.67	0.66	0.01
15	1	0.66	0.66	0.66	0.00
18	1	0.71	0.71	0.71	0.00
21	1	0.66	0.66	0.66	0.00
25	1	0.71	0.71	0.71	0.00
Means	6	0.66	0.76	0.69	0.04

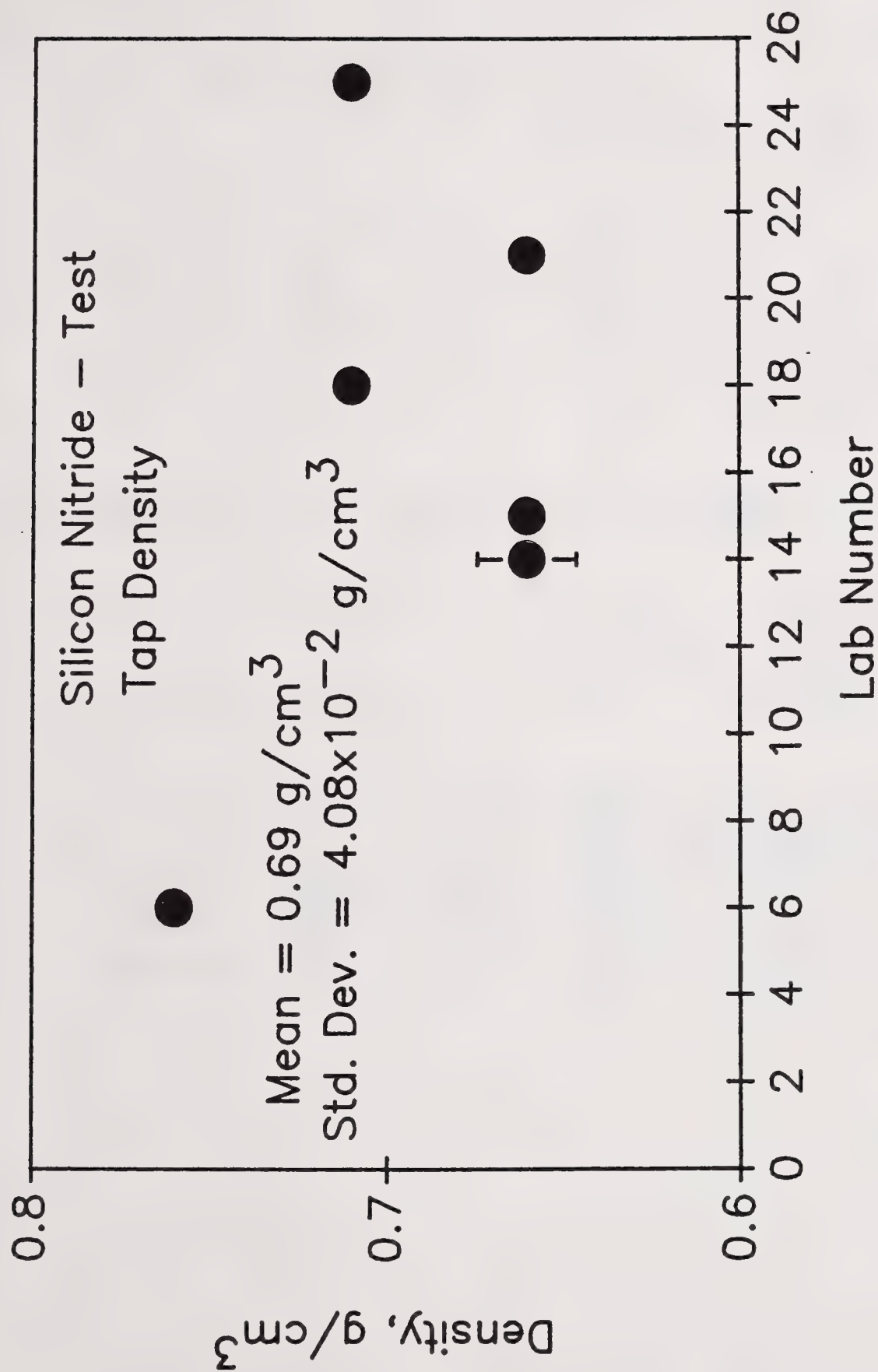


Figure 4 Tap density of silicon nitride (Test) powder reported by participating labs.

Table 2.2.3.3 Liquid Density in Hydrocarbon, Archimedes Method

Material: Silicon Nitride, Test Powder

Property: Liquid Density

Method: 6

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
25	1	0.43	0.43	0.43	0.00
Means	1	0.43	0.43	0.43	0.00

2.2.4. Bulk, Tap and Liquid Density of Silicon Carbide Powder

Table 2.2.4.1 Bulk Density by He Pycnometer

Material: Silicon Carbide

Property: Bulk Density

Method: 4

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
17	36	3.08	3.19	3.1459	0.0238
23	18	3.12	3.16	3.1468	0.0113
Means	2	3.15	3.15	3.1464	0.0006

Table 2.2.4.2 Tap Density

Material: Silicon Carbide (Figure 5)

Property: Tap Density

Method: 3

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
6	1	0.83	0.83	0.83	0.00
10	16	0.71	1.80	1.69	0.26
15	1	0.77	0.77	0.77	0.00
18	1	0.68	0.68	0.68	0.00
21	3	0.75	0.76	0.75	0.01
25	1	0.78	0.78	0.78	0.00
Means	6	0.68	1.69	0.92	0.38

Table 2.2.4.3 Liquid Density in Hydrocarbon, Archimedes Method

Material: Silicon Carbide

Property: Liquid Density

Method: 6

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
25	1	0.40	0.40	0.40	0.00
Means	1	0.40	0.40	0.40	0.00

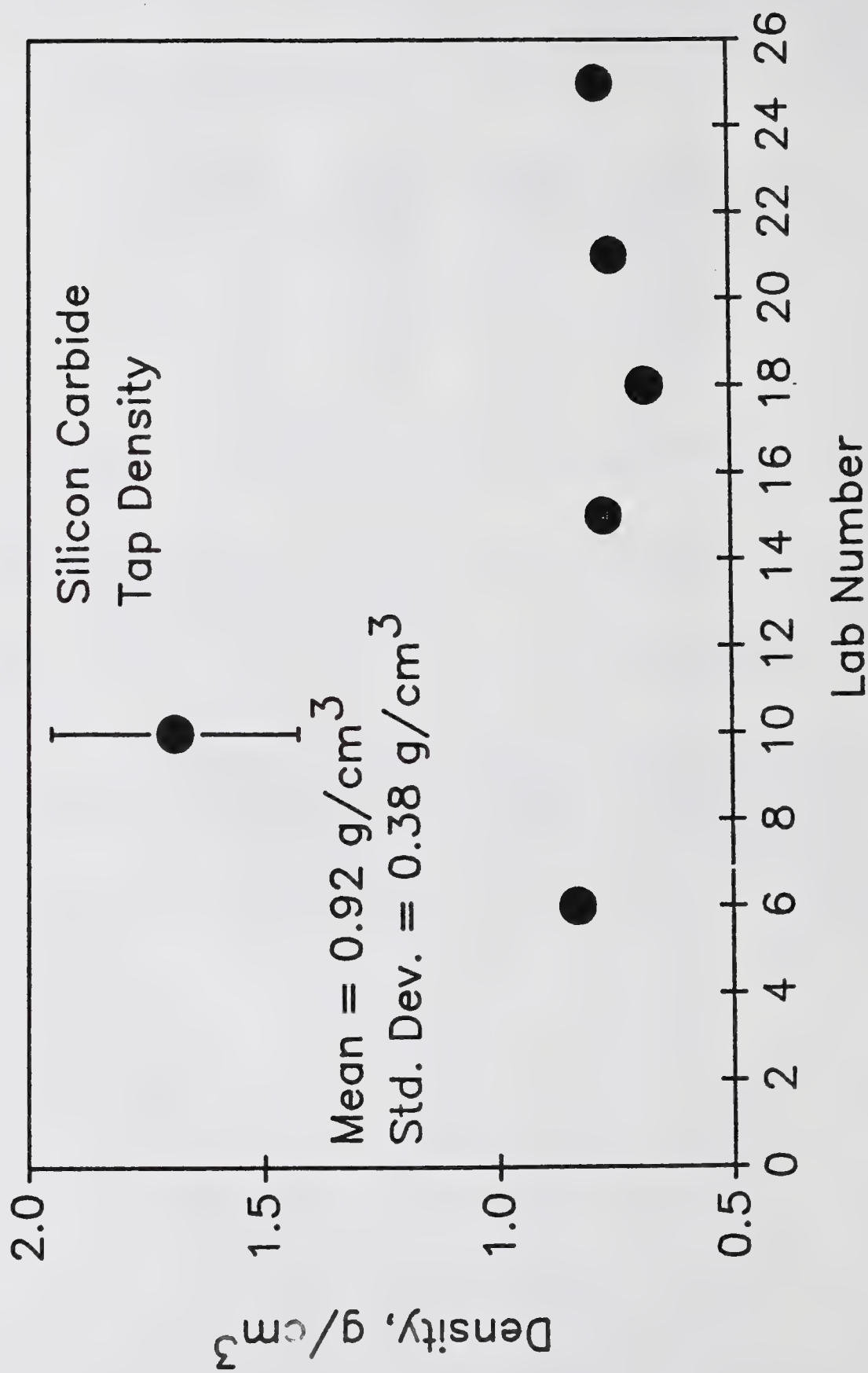


Figure 5 Tap density of silicon carbide powder reported by participating labs.

2.2.5. Bulk and Tap Density of Silicon Powder

Table 2.2.5.1 Bulk Density by He Pycnometer

Material: Silicon
Property: Bulk Density
Method: 4

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
17	40	2.300	2.350	2.3261	0.0142
23	17	2.328	2.344	2.3328	0.0049
Means	2	2.326	2.333	2.3295	0.0048

Table 2.2.5.2 Tap Density

Material: Silicon (Figure 6)
Property: Tap Density
Method: 3

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
6	1	1.08	1.08	1.08	0.00
15	1	1.03	1.03	1.03	0.00
17	3	1.46	1.48	1.47	0.01
18	1	0.78	0.78	0.78	0.00
Means	4	0.78	1.47	1.09	0.28

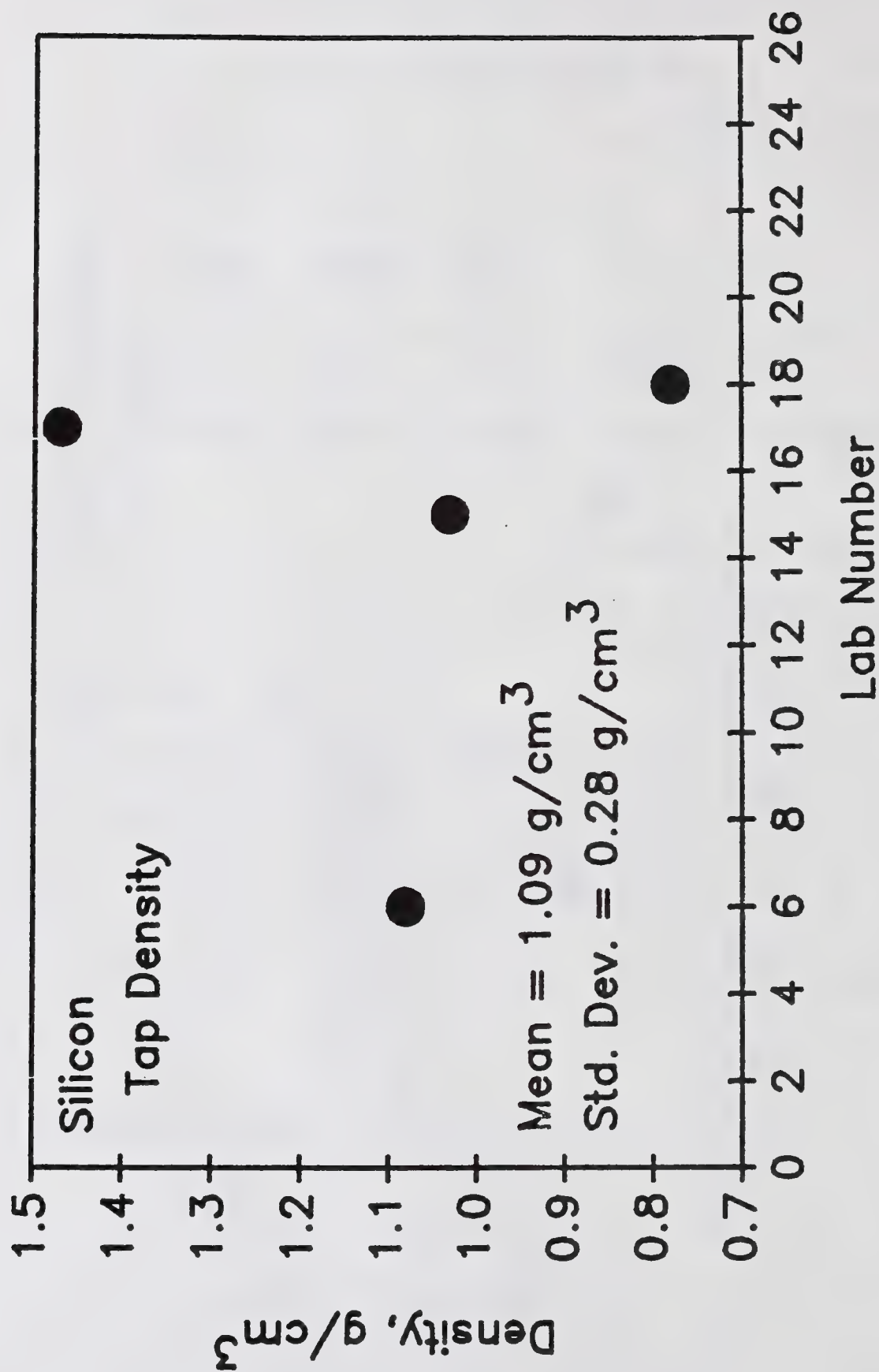


Figure 6. Tap density of silicon powder reported by participating labs.

2.2.6. Bulk, Tap and Liquid Density of Yttria-Zirconia Powder

Table 2.2.6.1 Bulk Density by He Pycnometer

Material: Zirconia
Property: Bulk Density
Method: 4

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
17	14	5.6060	5.8100	5.6870	0.0564
18	4	5.9030	6.0010	5.9480	0.0489
Means	2	5.6870	5.9480	5.8175	0.1846

Table 2.2.6.2 Bulk Density by Mercury Porosimeter

Material: Zirconia
Property: Bulk Density
Method: 13

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
22	6	6.0200	7.5660	6.7452	0.6493
Means	1	6.7452	6.7452	6.7452	0.0000

Table 2.2.6.3 Tap Density

Material: Zirconia (Figure 7)
 Property: Tap Density
 Method: 3

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
6	1	1.5200	1.5200	1.5200	0.0000
14	2	1.5000	1.5000	1.5000	0.0000
15	1	1.4900	1.4900	1.4900	0.0000
18	3	1.4600	1.4800	1.4700	0.0100
Means	4	1.4700	1.5200	1.4950	0.0208

Table 2.2.6.4 Liquid Density in Hydrocarbon, Archimedes Method

Material: Zirconia
 Property: Liquid Density
 Method: 6

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
14	2	1.2200	1.2400	1.2300	0.0141
Means	1	1.2300	1.2300	1.2300	0.0000

2.3. STATISTICAL ANALYSIS, R. Munro, NIST

The bulk density of a powder refers to the average mass per unit of actual volume of the particles in the powder. The total mass of a powder specimen can be measured with high accuracy using a microbalance. The helium pycnometer technique is most commonly used to determine the actual volume of the particles in the powder. The helium gas penetrates into the open pores and interparticle regions so that the volume of the gas displaced by the specimen is the sum of the volumes of the individual particles including contributions from regions of closed porosity. While only two laboratories reported measurement results, the method appears to be among the more precise and reproducible measurements made in this study, except for its application to zirconia. The results in Figure 8 indicate an unusually good agreement among laboratories.

In contrast, the tap density measurements, Figure 9, exhibit a large variance between laboratories, as well as being less precise. This result is not surprising considering the extent to which the procedural details in this technique can influence the measured value. The tap density refers to the average mass per unit of occupied volume and is determined by dividing the total mass of the specimen by the volume occupied by the powder in a graduated cylinder after being mechanically tapped a given number of times. The powder settles or compacts as the cylinder is tapped but retains a significant pore volume. The amount of interparticle pore volume varies with both the magnitude of the impact imparted by the tapping and the frequency of the tapping.

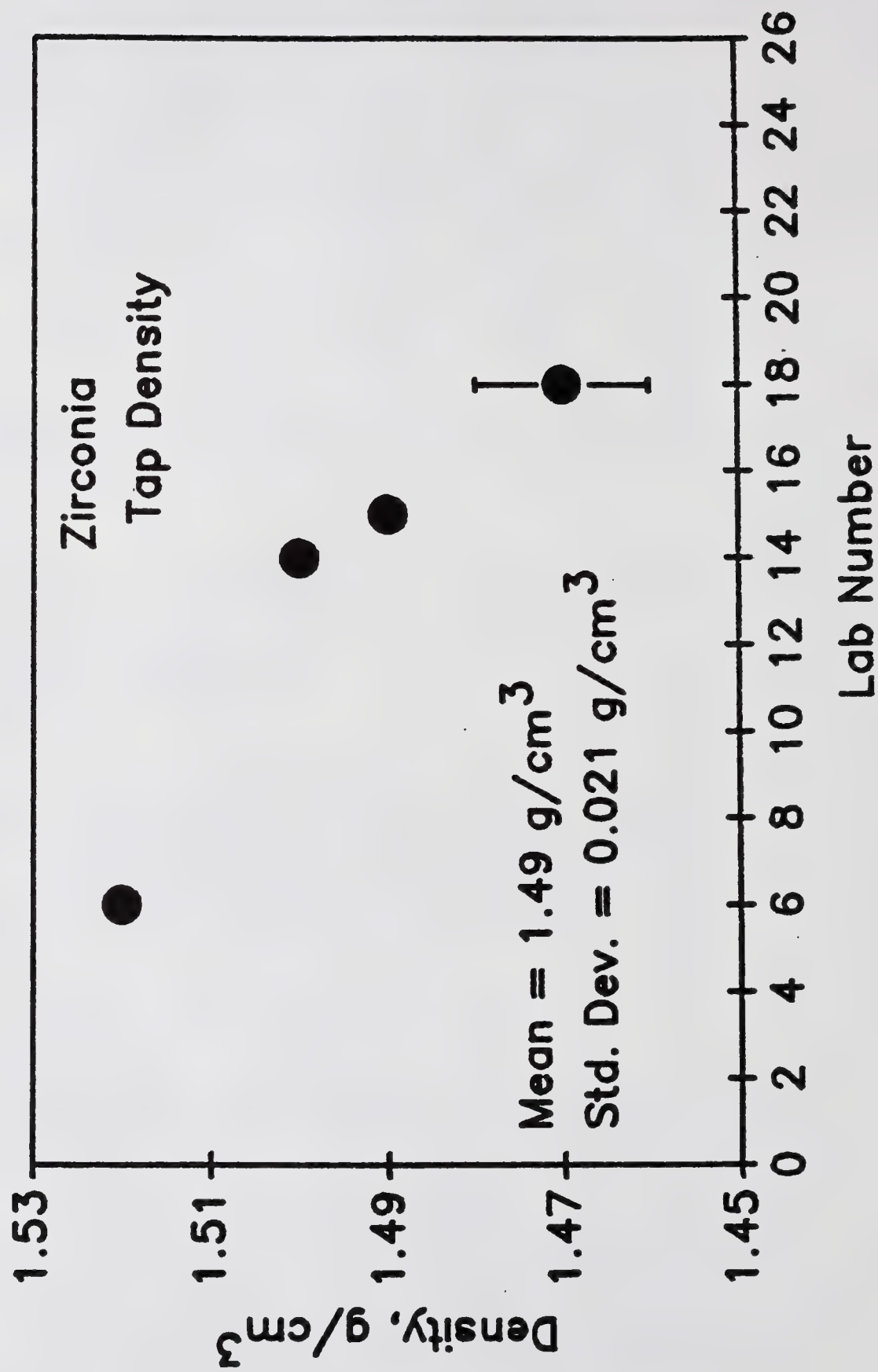


Figure 7. Tap density of yttria-zirconia powder reported by participating labs.

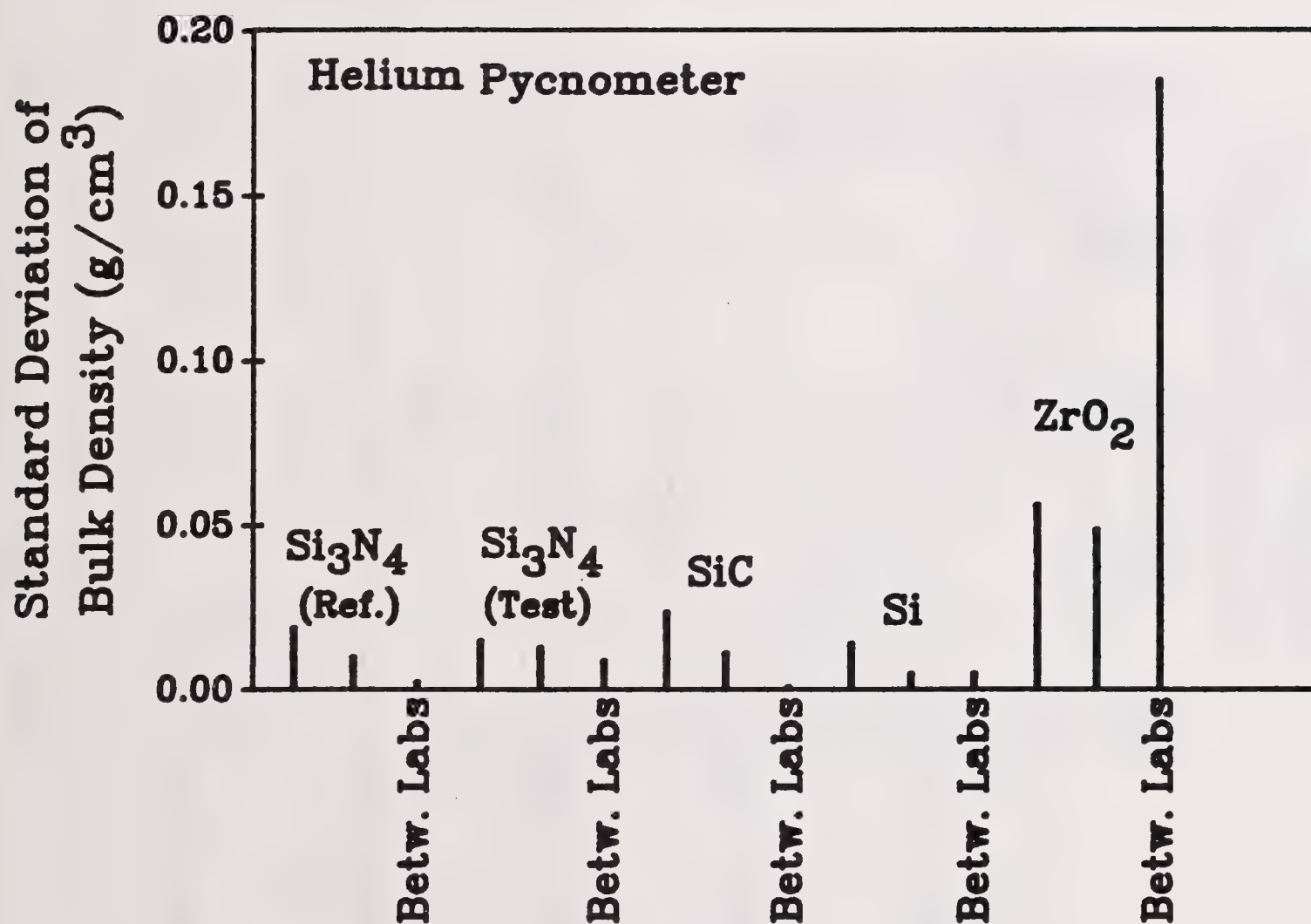


Figure 8. Within lab and between lab standard deviations in the measurements of bulk density for five powders as measured by various laboratories using the helium pycnometer method.

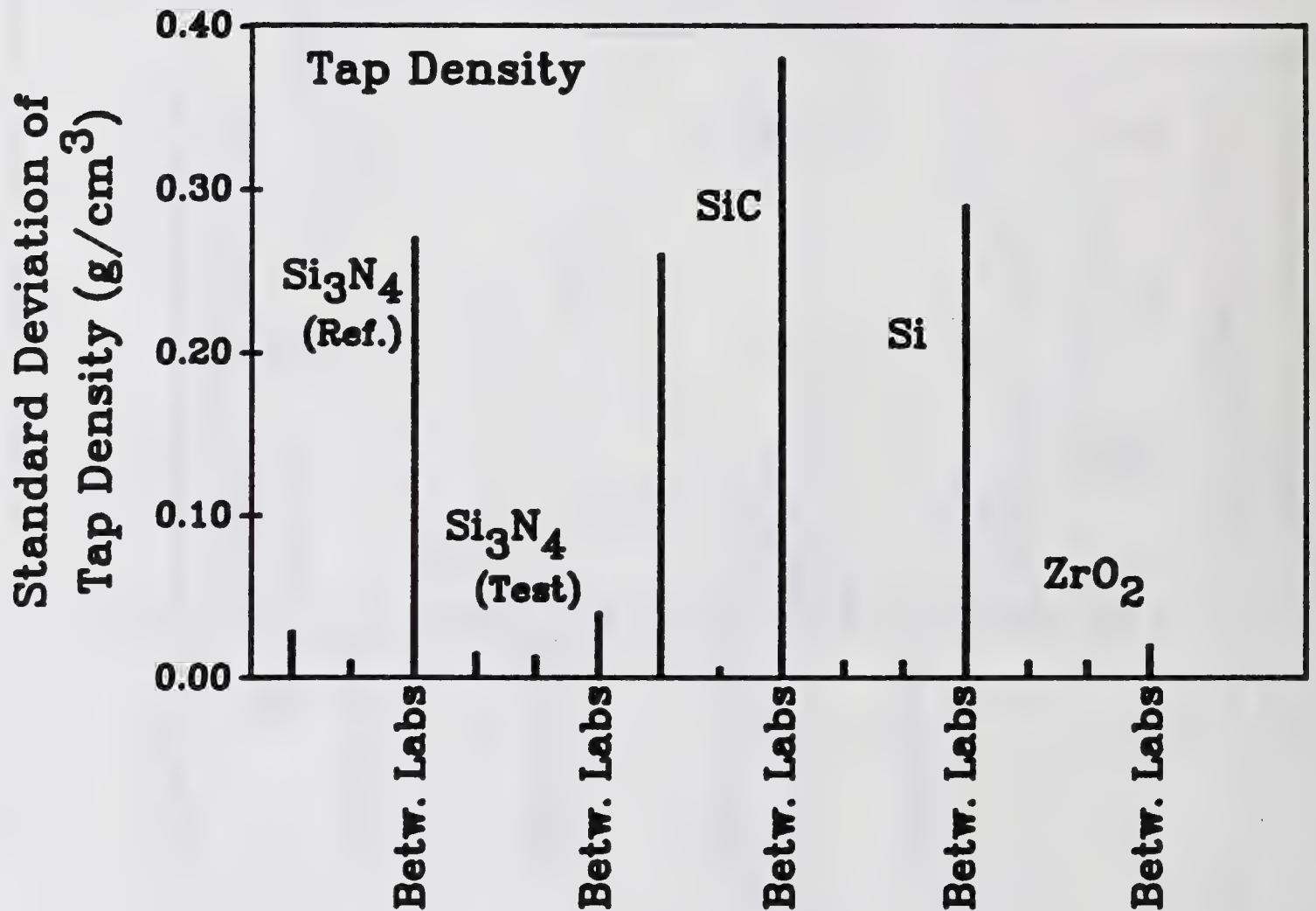


Figure 9. Within lab and between lab standard deviations in the measurements of tap density for five powders as measured by various laboratories using tap method.

3. SPECIFIC SURFACE AREA

R. Pompe, Swedish Ceramic Institute; S. G. Malghan, NIST

3.1. MEASUREMENT METHODS

3.1.1. Brunauer-Emmett-Teller (BET) Method

Use and Significance

The specific surface area of powders composed of deagglomerated, compact particles with a regular size and narrow size distribution will be closely related to the mean particle size. For other powders, the measured specific surface area will include factors such as shape factor, degree of agglomeration and internal porosity of the particles. Significant indication of particle regularity, internal porosity and agglomeration may be gained by combining surface area data with results of measurements of particle size.

Principle

The specific surface area measurement is usually based on the well-known BET theory which describes the multilayer adsorption of a gas upon a clean and uniform surface. The BET theory generalizes an older Langmuir monolayer theory by assuming that a layer of adsorbed molecules provides the sites for the adsorption of the next layer and that only local forces exist between the layers.

Instrument and Experimental Parameters

The experimental parameters which can influence the measurement are:

- sample handling
- sample quantity
- degassing temperature
- degassing time
- adsorbate gas
- other factors, such as standards, reference materials.

To avoid biased measurements, the powder to be analyzed should be sampled at many locations within the material. This is best done with a small spinning riffler. If a riffler is not available, the powder can be poured into a shallow pile or a long row and sampled at many points. Grab samples from a single location in the powder should not be used.

Typical amounts of powder used for samples were in the range of 1-5 grams. Some laboratories used amounts down to 0.1-0.5 grams and still obtained precision in specific surface area values within the range of data obtained by other labs.

Degassing depends on the pressure, temperature and duration at which the surface is conditioned. Degassing conditions differed depending on the powder

and laboratory practice. One lab (No. 22) also carefully checked outgas rate. Degassing conditions are summarized in Table 3.1.1.

Table 3.1.1. Summary of Instrumental and Experimental Conditions in the Single(s) and Multi-Point(M) BET Measurements

Lab No.	BET Meas.	Degass time (h)	T(°C)	Adsorbate gas	Instrument type
1	M/S	4	150/vac	N ₂	Micromeritics,
2	M			N ₂	Digisorb 2600
3	M/S	4-8	110 (+RT/vac)	N ₂	
4	S	1-3	/vac	N ₂	Quantachrome, Monosorb
5	M	2	215/vac	N ₂	Quantachrome
6	S		RT-288	N ₂	Quantachrome
7	M/S	20 min	215	N ₂	Flowsorb II 2300,
8	M	17	200	N ₂	
10	M	18	220	N ₂ /Kr	Digisorb 2500
		(1-2)	(-300)		
11	S		350	N ₂ /Kr	Quantasorb. + Accusorb.
12	M			N ₂	Digisorb 2600
13*	S	40 min (air)	300	N ₂	Stroehlein AREA meter II (DIN 66132)
14	S	15/20 min	100/150-200	N ₂	a) - " -
	S	15/1	100/200/vac	N ₂	b) Micromer. 2400
15	S		100/150-200(N ₂)	N ₂	Stroehlein
16	S			N ₂	- " -, DIN 66132
17	S	2-3	150	N ₂	Sorpty 1750, C Erba Inst.
18	S	0.5-4	350	N ₂	Microm. 2200 + Desorb 2300 A
19	S	1	200-300	N ₂	Desorb 2300
20	S/M		140	N ₂	(N ₂ /He dynamic flow)
21	S	2	250	N ₂	Quantasorb, Quantachr.
22	S			N ₂	Flowsorb 2300
23	M			N ₂	Monosorb, Quantachrome
24	M	2	250	N ₂	Quantasorb

*ZrO₂: 110 °C/16 h, heating in air

The measurements are accomplished either by a simplified singlepoint run at the saturation vapor pressure of the liquified adsorbent gas or by the more detailed multipoint technique. In the latter case, runs are made at different adsorption pressures, and the specific surface area is calculated from the slope and intercept of a straight line usually obtained from a BET plot of

adsorption-to-saturation pressure ratio in the range 0.05-0.25. In most cases three runs are used to determine the specific surface area. The measurements are performed either under static or dynamic gas flow conditions. For example, flowing N₂/He gas mixtures are frequently used for dynamic conditions. Two labs (No. 8, 22) used Kr gas as an adsorbate which gave lower values as compared to the results with N₂ adsorbate gas.

Computer automation of the measurement enables a determination of the adsorption isotherm.

Calibration and Standards

The use of the manufacturer's recommended procedure generally yields highly reproducible data. However, calibration of apparatus against standard equipment at regular intervals and/or calibration with standard powders should decrease disparities.

A recommended general procedure is given by ASTM C1069-86, Standard Test Method for Specific Surface Area of Alumina or Quartz by Nitrogen Adsorption.

For single point determinations consult:

DIN 66132. Determination of specific surface area by absorption of nitrogen according to the single-point differential method of Haul and Dumbgen.

Alpha alumina powders certified by the National Physical Laboratory, Teddington, U.K., and meeting the ISO conditions for certified standard powders are available from the Community Bureau of Reference of the European Economic Community or, in the United States from the National Institute of Standards and Technology as Standard Reference Materials (SRM's) 8005 to 8008. These references cover the range 0.1 to 2.1 m²/g. NIST also offers SRM 8570, a calcined kaolin, produced in conjunction with the ASTM, with a specific surface area of 10.89 m²/g.

Despite the availability of external standards, only a few laboratories in this study reported the use of standards. In some cases the reported standards were those internal to the practices of that laboratory or those furnished by the manufacturer; for example, lab No. 22 reported the use of two TiO₂ standards with specific surface areas of 24.3 ± 0.6 and 10.3 ± 0.3 m²/g.

Instrumental Effects

The instrument information has been taken from Table 3.1.1 which is based on primary data as provided by all labs. Several labs did not give sufficient information on their instruments. The instruments selected include only those used by two or more laboratories for three or more types of powders.

The instruments compared for single point measurements are Quantasorb, Desorb 2300 and Stroehlein, and for multipoint measurements the instruments are Quantasorb and Digisorb 2500/2600. The total number of data collected per instrument varies considerably; from 50 to 100 values for Quantasorb and 1 to

10 for Stroehlein. The primary data base is thus not fully satisfactory for all instruments - none the less, it seems apparent that none of the instruments appear to give data sets which systematically diverge from each other. Hence, the variation of the data obtained seems to depend more on other factors than the selection of a particular instrument.

Lab. 10 noticed that by switching from stationary gas adsorption to the flow-type mode, the BET single point data for LC 10 Si_3N_4 powder decreased by approximately 5%.

Relatively coherent data were obtained from different laboratories even for the low surface area Si powder. This indicates an overall high absolute instrumental accuracy.

3.1.2. Results and Discussion

In this section, a general comparison is made with respect to the number of laboratories providing data, number of measurements made, and the spread of data when all the data are considered. In the ensuing subsections an attempt is made to evaluate selected data with respect to instrumental and experimental parameters, the internal consistency of powders and specific features of a powder.

General Features

Most laboratories provided single point data, but several supplied both single and multi-point values. Typically, in more than 50% of the cases the number of measurements per sample was sufficient to permit calculation of a mean value. Table 3.1.2 shows the number of laboratories providing data for single and multi-point measurements, respectively, for different powders investigated.

Several laboratories supplied only 1 or 2 data. These values are part of their particular data population. A reliable comparison of these data with average values of other laboratories cannot be made because the precision of such data cannot be estimated. However, it still may be of interest to include these data in a graphical representation (see Figures 9-18).

Table 3.1.2. Number of Laboratories Single and Multipoint BET Measurements Reported Per Powder

Powder	Single-point	Multi-point
Si ₃ N ₄ ref.	14	10
Si ₃ N ₄ test	12	8
SiC	11	9
Si	11	5
ZrO ₂	10	7

The relative standard deviations for mean values calculated from all data for both single and multi-point values were in the range of 5-10% (see Table 3.1.3). In absolute terms, the standard deviation of the mean was typically approximately equal to less than 1 m²/g. The results for Si gave a standard deviation of 0.2 m²/g and almost an order of magnitude lower value for the specific surface area. The multi-point results of one lab (No. 22) affected strongly the mean value and standard deviation for Si. The spread of the values of the population of all data for a powder was generally of the same order of magnitude as that for the spread of data obtained by particular laboratories. In some cases the variation for a powder was significantly larger than the within laboratory variation. This will be considered later in the analysis.

The average values for the single and multi-point results, where appropriate with the standard deviations, are shown in Figures 9-18.

Table 3.1.3 Average Values and Standard Deviations for Single and Multi-point BET Results

Powder	Single-point			Multi-point		
	Mean m ² /g	Standard Deviation m ² /g	%	Mean m ² /g	Standard Deviation m ² /g	%
Si ₃ N ₄ ref.	13.11	0.72	5.5	13.51	0.62	4.6
Si ₃ N ₄ test	9.43	0.61	6.5	9.81	0.52	6.3
SiC	13.34	1.12	8.4	14.53	0.96	6.6
Si	1.92	0.2	12.0	2.27	0.61	26.9
ZrO ₂	16.84	1.4	8.3	18.36	0.59	3.2

Single-point vs Multi-point Data

In Table 3.1.3 it can be seen that the average multi-point values based on all data for each powder are always higher by 2-10% than the corresponding average single point values. However, for those laboratories which measured the specific surface areas using both methods (Lab 14 for SNR; see Tables 3.2.1.1.

and 3.2.2.2; Labs 14 and 20 for SNT; see Tables 3.2.2.1 and 3.2.2.2) the single-point values were slightly higher than the multi-point values. In the case of Lab 14, some samples from the same vials were used for both test, with single-point values higher than multi-point values.

The multi-point specific surface areas are determined from the BET isotherm. A typical plot (Lab 1) of the volume of nitrogen gas adsorbed vs relative pressure is shown in Figure 10a for silicon powder. Figure 10b shows a corresponding BET plot of $1/VA(P_0/P-1)$ against the relative pressure (P/P_0) for the same data.

There may be several reasons for the divergence between the single point and multi-point results:

- (a) statistical - the limited population of the participating laboratories may be subject to a fluctuation, giving rise to a systematic difference;
- (b) experimental/instrumental - somewhat differing conditions between the two types of measurements may have been significant;
- (c) algorithm-related - the multipoint values are computed by a regression analysis; or
- (d) virtual - the multi-point values may be closer to the true surface area.

The correlation coefficients obtained in the multi-point analyses were typically 0.995 and higher. Nonetheless, as indicated later in this discussion, the multi-point values obtained by laboratories which performed a large number of runs on one sample displayed generally larger mean deviations compared to corresponding single- point data.

Several laboratories which measured the surface areas in the multi-point mode also determined the Langmuir surface areas (e.g., Lab 6). These surface areas were typically greater than those calculation from the BET equation (e.g., Lab 1).

For a general assessment as to which method to employ, the experimentalist must consider which method gives the most reproducible values, and which technique is easier to utilize from the experimental/instrumental point of view.

Internal Consistency of the Powder Samples

The issue of concern of this subsection is how the variation of data may have been influenced by the riffling procedure as evidenced by variation between different vials and as compared to variation in independent measurements made on one vial of powder. To identify the various contributions to the variance of the measurement requires independent analyses of replicate vials, analysis of replicate subsamples from each vial and replication of the measurement process. "Independent analyses" means that all aspects of the analysis of

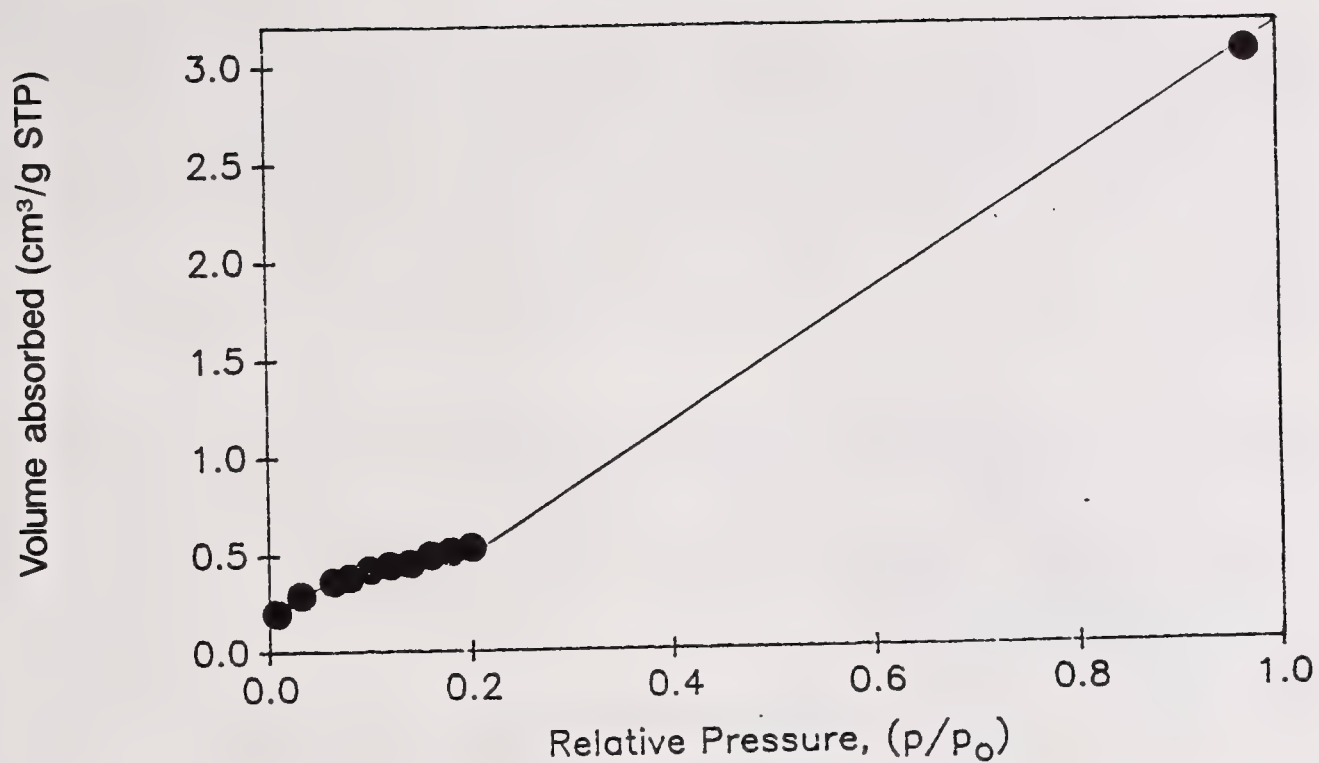


Figure 10a. Plots of BET multipoint data: volume of gas adsorbed vs. relative pressure.

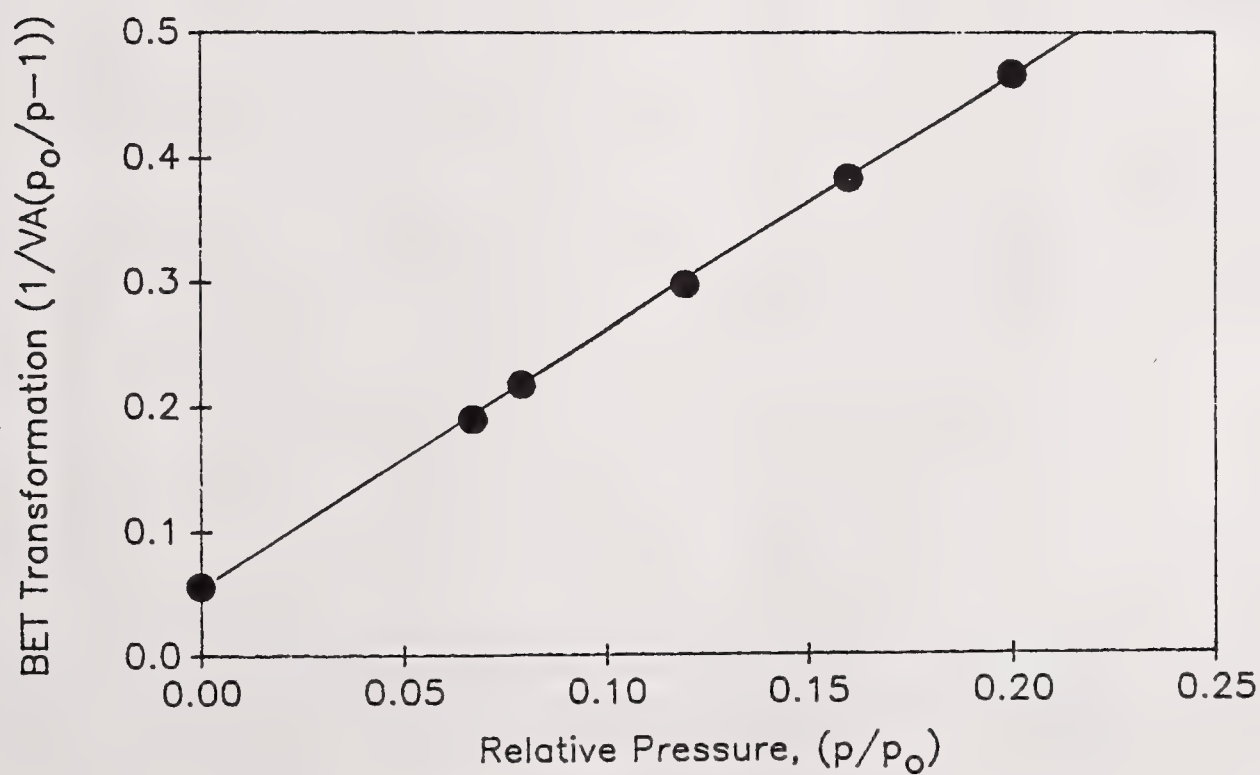


Figure 10b. Plots of BET multipoint data: BET transformation of adsorption data in a.

separate analytical samples are carried out in the same manner. Multiple instrumental readings on the same sample do not constitute independent measurements, albeit they do yield an estimate of the variance assignable to the instrumental readings. The tabulation of data in Chapter 3.3 does not make a distinction between multiple instrumental readings and independent measurements in which replicate subsamples of a sample were prepared and analyzed. Furthermore, the raw data furnished by a laboratory often did not clearly indicate whether measurements identified as replicate runs were truly independent. In a few cases sufficient information was provided to enable independent runs and multiple readings to be distinguished.

Most laboratories which reported more than one measurement only analyzed one sample from a sample vial and reported only one instrumental reading. Table 3.1.4 gives the means and standard deviations for laboratories which reported more than 10 determinations, either multiple readings or replicate subsamples, per vial and which analyzed 2 or more vials. The powders included in Table 3.1.3 are Si_3N_4 Ref., Si_3N_4 Test and SiC. Si powder was not included because it had a low absolute surface area;

Table. 3.1.4 Mean and Standard Deviation for Analyses with Replicate Sample or Multiple Instrumental Measurements.

Powder	Lab No.	Single(s)/ Multi-point(m)	Mean, m^2/g	Standard Deviation
Si_3N_4 Ref.	4	s	13.39	0.32
	17	s	12.97	0.27
	22	m	13.51	1.03
	23	m	13.40	0.28
Si_3N_4 Test	4	s	9.79	0.03
	8	s	10.28	0.06
	17	s	9.01	0.23
	9	m	9.73	0.32
	23	m	9.65	0.12
SiC	3	s	12.71	0.19
	4	s	13.72	0.05
	17	s	13.04	0.07
	9	m	14.63	0.13
	23	m	14.27	0.18

also, the data showed low variation for the few laboratories which made a sufficient number of runs. The YSZ powder was not included because of a degassing dependence in the measurements was apparent in the data.

With the exception of Lab 22, the standard deviation was within 0.3% - 3.3% of the mean value. Lab 22 carried out measurements using N_2 and Kr so that the relatively large standard deviation (7.6% of the mean) includes the differing

effects of the two adsorbates. For the other laboratories the variances generally contain the contributions from the preparation of the IEA samples (riffing error; standard deviation, s_R), from the extraction and preparation of analytical subsamples (preparation error; standard deviation, s_P) and from the instrumental measurement (measurement error; standard deviation, s_M). The total variance can be expressed as

$$s^2 = s_R^2 + s_P^2 + s_M^2 \quad (2.1)$$

For the laboratories reporting a large number of determinations of specific surface area, the manner in which those measurements are distributed with respect to IEA samples, subsamples and measurements is indicated in Table 3.1.5.

It is evident from Table 3.1.5 that the effect of taking subsamples was not adequately tested. With the exception of Lab 17, the subsampling effect is confounded with the run effect. The results for the SNR powder provided by Lab 23 could not be evaluated due to a large uncertainty in measurements for one of the two subsamples from IEA sample SNR 1471. An analysis of variance of the results of Lab 17 for SNR, SNT and SiC powders is given in Table 3.1.6.

Table 3.1.5 Distribution of Measurements with respect to Samples, Subsamples and Runs.

No.	Method	No. of Data	No. of Samples	No. of Subsamples per Sample	No. of Lab Runs per Subsample
<u>SNR</u>					
4	S	15	5	1	2-4
17	S	80	16	#582, 2 #1381, 3 All others, 1	4-5
18	S	9	1	3	3
23	M	24	5	#1471, 2 All others, 1	3
<u>SNT</u>					
17	S	38	6	#312, 2 #938, 2 #1419, 2 All others, 1	4-5
18	S	9	1	3	3
9	M	20	5	1?	4
23	M	24	7	#116, 2 All others, 1	3
<u>SiC</u>					
17	S	33	7	#154, 3 All others, 1	4-5
18	S	9	1	3	3

Table 3.1.6 Estimates of the Standard Deviation and Its Components for the Results of Lab 17. Units are m^2/g .

Powder	Data Used in the Analysis	Standard Deviation				
		(1)	(2)	(3)	(4)	(5)
SNR	All data	0.3196				
	Subsample av. and s.d.		0.3260	0.0876		
	#582, #1381 subsample av.			0.0701	0.0900	
	Estimate, using Col. (2) and (4)					0.300
SNT	All data	0.2304				
	Subsample av. and s.d.		0.2349	0.0694		
	#312, #938, #1419 subsample av.				0.0364	
	Estimate, using Col. (2) and (4)					0.232
SiC	All data	0.1989				
	Subsample av.		0.1697	0.1381		
	Insufficient data for reliable estimate of s_p and s_R . and s.d.					

Standard Deviation Columns above

Col. (1) Total standard deviation, s .

Col. (2) $[s_R^2 + s_p^2]^{1/2}$

Col. (3) s_M , run standard deviations pooled over all subsamples.

Col. (4) s_p .

Col. (5) s_R .

The estimates for s_R are 0.30 m²/g for the SNR powder and 0.23 m²/g for the SNT powder. The SiC value could not be estimated. Although s_R cannot be estimated for the other laboratories and for the SiC powder, the sum

$$s_S^2 = [s_R^2 + s_P^2] \quad (2.2)$$

of the variances can be estimated. For other laboratories, values of the standard deviation, s_S , were in the range 0.17 to 0.35; for the SNR powder, 0.09 to 0.17, for the SNT powder. For the SiC powder, for which only data from Lab 17 were available, s_S was 0.065. Using the formula

$$2[s_S^2/2\hat{a}_S + s_P^2/2\hat{a}_P]^{1/2}$$

where $\hat{a}_S$ is the number of degrees of freedom in estimating combined variance and $\hat{a}_P$ is the number of degrees of freedom in estimating s_P^2 , the approximate confidence range of s_R obtained from the results of Lab 17 can be estimated. For the SNR powder, the range is approximately 0.15 to 0.45; and for the SNT powder, 0.11 to 0.35. Thus, the values of s_R as suggested by the results for s_S for all laboratories are consistent with the estimate obtained from Lab 17 in the case of the SNR powder, but possibly lower than the estimate in the case of the SNT powder.

The range of estimated relative sample error due to riffing is 1.0 to 3.5% for the two Si₃N₄ powders. This range can be compared with the estimated maximum error of 0.42% obtained for sampling of a 60:40 coarse:fine sand mixture.⁵ The source of the increased error for the Si₃N₄ cannot be definitely established; however, the poor flow characteristics of these powders is suspected. Furthermore, it is to be noted that the surface area values are roughly uniformly distributed with no significant outliers apparent.

The measurement error for the multi-point determinations appears to be about twice that for the single-point determinations. The riffing error may be comparable to the measurement error obtained by the multi-point method. Based on the results of Lab 17, which riffled the IEA sample to obtain subsamples, the sample preparation error for the Si₃N₄ may be small. These statements are at best tentative since the replication of samples, subsamples and measurements was inadequate for the calculation of accurate estimates.

Specific Features of the Data

SNR and SNT

See above discussion of internal consistency of the powders. (Figures 11-14)

SiC

Lab 1 stressed that samples had to be mixed thoroughly to obtain reproducible results - i.e., the samples were not homogeneous. The SiC data show the relatively largest mean deviation (both relative and absolute values). Unfortunately, only Laboratories 9 and 23 (multi-point) and Labs 3, 4, 17 and 18 provided sufficient amounts of data for computing reasonable, within

laboratory, mean values. These mean values show a relatively narrow spread (see Figures 15-16).

Si

A relatively narrow spread of data was obtained in spite of the low BET surface area of this powder (see Figures 17-18). Values within the deviation range were obtained even when small amounts of powder were used; e.g., Lab 23 used 0.17 to 0.40 g of powder. Regarding the large spread for the multi-point data reported by Lab 22, the data contain measurements by N₂ and by Kr adsorbate gases. Surprisingly, the BET areas obtained with N₂ were about twice those obtained with Kr.

YSZ

SEM and TGA indicated that the powder was spray dried and, thus, contained traces of a binder which was removable only at high degassing temperatures or long times. In addition, this material is known to be fairly hygroscopic. Lab 18 demonstrated a considerable effect of degassing temperature on the BET area (multi-point):

Effect of Degassing Temperature on Specific Surface Area

	1st Run	2nd Run

Room Temp.	16.2	17.1 m ² /g
55°C	17.4	18.3
288°C	18.9	19.8

Lab 4 showed that the BET area (single-point) increased as the degas time at 350°C increased from 0.5 to 4 hours. The data shown in Figure 19 seem to fall into two groups consistent with the extent of degassing. Those data provided by Labs 4, 16 and 18 were obtained at high degas temperatures and at times typically not less than 2 hours. The multi-point data are shown in Figure 20.

Conclusions

The relative standard deviation for the average value for each powder, calculated using all data, was in the range of 5-10%, except for Si which had a low surface area.

The multi-point average values were higher by 3-10% than the corresponding single-point values. From the available data, it was not possible to deduce whether this difference is real. Two of the laboratories which performed measurements by both methods obtained higher single-point data.

For the SNR, SNT and SiC powders an average value obtained within an uncertainty band of $\pm 1 \text{ m}^2/\text{g}$ around the average values shown in Table 3.1.3 can be expected to be acceptable. For the Si powder the band may be set at $\pm 0.5 \text{ m}^2/\text{g}$.

No significant difference in average values or spread of data was found when comparing laboratories which used different types of instruments. The relatively small spread of absolute values for the Si powder with low specific surface area indicated a good instrument-to-instrument measurement capability.

The vial-to-vial spread of average values was significantly higher, in particular for Si_3N_4 Ref. and SiC, than for runs on several samples from one vial. The variation within one vial was on the same order of magnitude as that for several runs in which each run used a single sample from a single vial. Estimates of the riffing error are in the approximate range of 1.0 to 3.5% for the two Si_3N_4 powders. However, part of the apparent riffing error may be due to vial-to-vial variation in apparent surface area due to formation of closed pore agglomerates. The presence of closed pore agglomerates may also account for the observed vial-to-vial density variation for these powders.

For measurements performed several times on a single sample, the standard deviation for the multi-point measurements was frequently larger than for corresponding single-point measurements.

The measurement of specific surface area for the ZrO_2 powder was suggested to be considerably dependent on the degassing conditions.

Suggested future activities:

- ZrO_2 - interlaboratory measurements using a fixed degassing temperature and time;
- A statistically balanced set of interlaboratory measurements should be performed on one powder, such as the Si_3N_4 powder, with each laboratory using multiple subsamples of each vial and multiple independent instrumental measurements on each subsample;
- Measurement of density, BET area, particle size distribution (plus SEM examination) should be carried out on one sample taken from each vial exhibiting large data variation.

3.2. STATISTICAL DATA

3.2.1. Silicon Nitride Reference (SNR) Powder

Table 3.2.1.1 Single-Point Determination

Material: Silicon Nitride, Reference Powder

Property: SINGLEPT

Method: 1

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
2	1	13.20	13.20	13.20	0.00
3	3	11.60	11.80	11.67	0.11
4	15	13.10	13.90	13.39	0.32
5	1	12.40	12.40	12.40	0.00
8	4	14.00	14.04	14.02	0.02
11	1	13.70	13.70	13.70	0.00
14	5	13.60	14.80	14.18	0.44
15	2	12.90	13.10	13.00	0.14
16	7	13.60	14.00	13.76	0.14
17	80	12.44	13.71	12.97	0.27
18	9	12.45	13.04	12.87	0.19
24	5	12.00	12.60	12.22	0.23
25	3	13.10	13.20	13.13	0.06
Means	13	11.66	14.18	13.12	0.72

Table 3.2.1.2 Multi-point Determination

Material: Silicon Nitride, Reference Powder

Property: MULTIPT

Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	2	14.20	14.51	14.35	0.21
5	1	13.20	13.20	13.20	0.00
6	1	12.93	12.93	12.93	0.00
7	2	13.47	13.56	13.51	0.06
9	5	13.72	15.35	14.72	0.65
13	10	11.29	14.61	12.95	0.82
14	4	12.30	13.10	12.72	0.38
20	7	12.92	13.48	13.21	0.21
21	2	13.59	13.61	13.60	0.01
22	14	12.07	15.53	13.51	1.03
23	21	12.75	13.89	13.40	0.28
Means	11	12.72	14.72	13.46	0.60

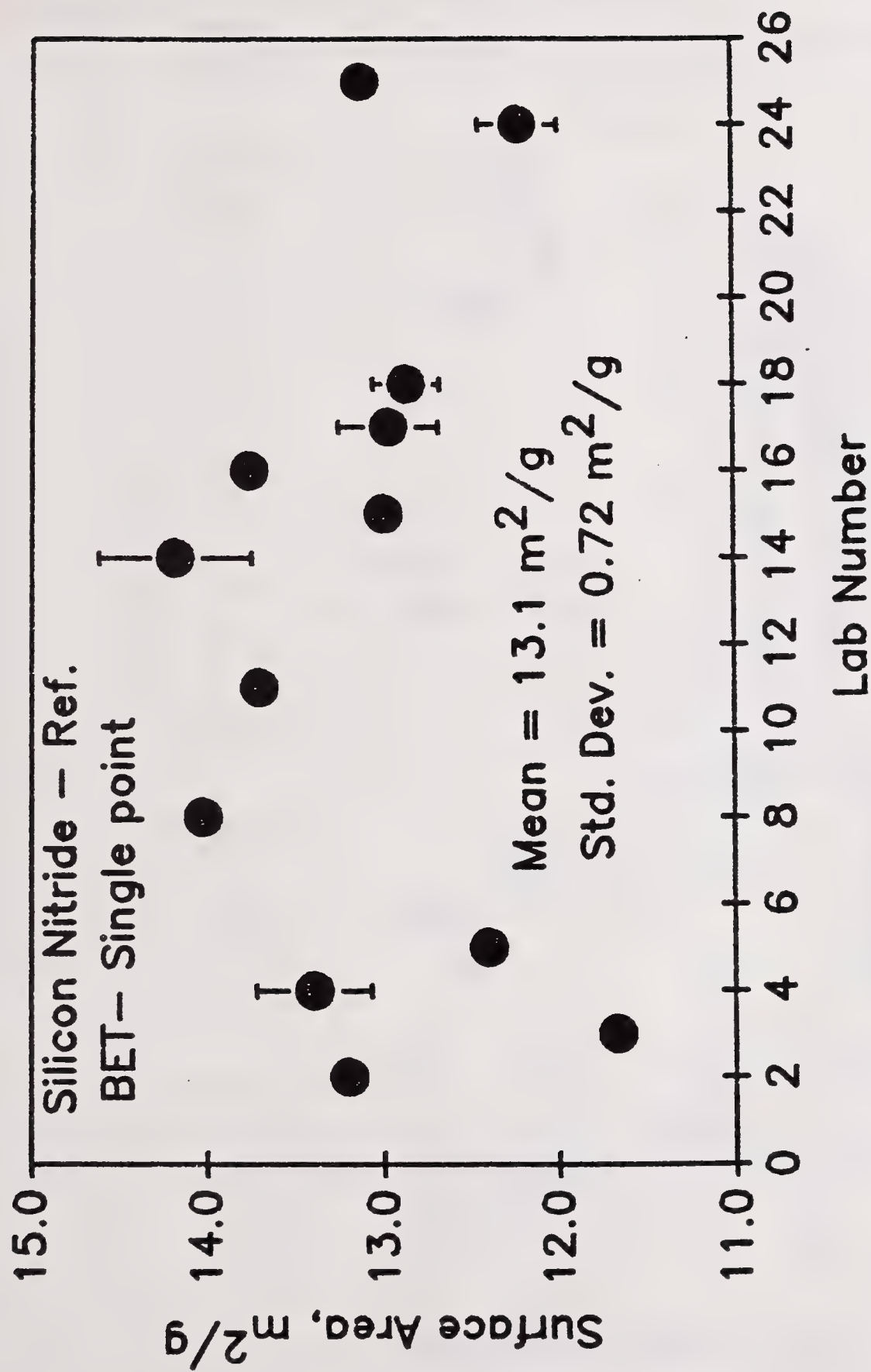


Figure 11. Specific surface area of SNR powder as reported by participating labs using single point BET method.

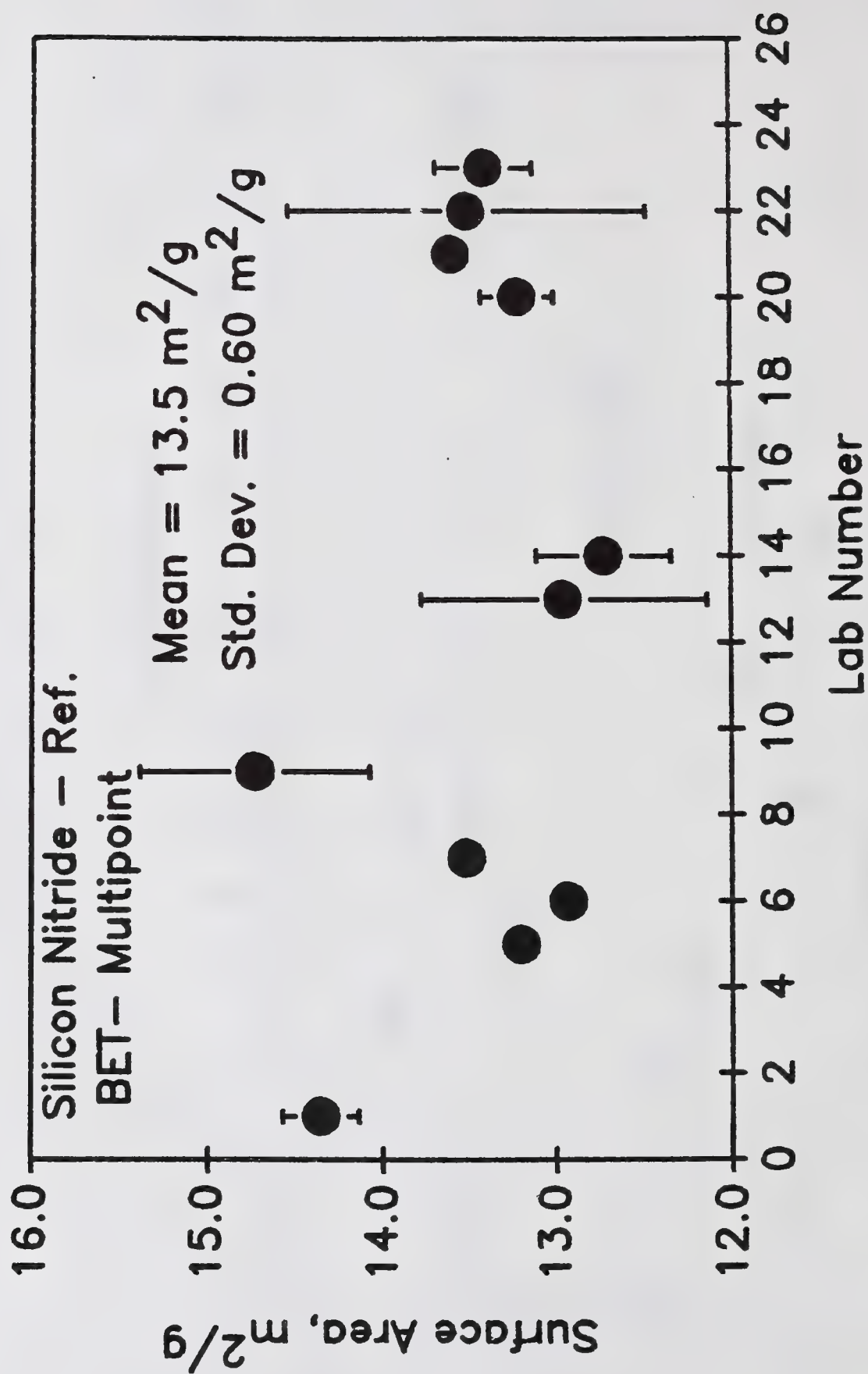


Figure 12. Specific surface area of SNR powder as reported by participating labs using multipoint BET method.

3.2.2 Silicon Nitride Test (SNT) Powder

Table 3.2.2.1 Single-Point Determination

Material: Silicon Nitride, Test Powder

Property: SINGLEPT

Method: 1

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
2	2	9.10	9.10	9.10	0.00
3	2	8.44	8.49	8.46	0.03
4	12	9.72	9.83	9.79	0.03
5	4	9.10	9.20	9.15	0.05
8	10	10.21	10.43	10.28	0.06
14	3	9.00	11.00	10.33	1.15
15	1	8.70	8.70	8.70	0.00
16	6	8.88	9.29	9.10	0.18
17	38	8.62	9.48	9.01	0.23
18	9	9.21	9.83	9.49	0.17
20	2	10.00	10.15	10.07	0.10
25	2	9.53	9.55	9.54	0.01
Means	12	8.46	10.33	9.42	0.60

Table 3.2.2.2 Multi-point Determination

Material: Silicon Nitride, Test Powder

Property: MULTIPT

Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
6	1	9.31	9.31	9.31	0.00
7	6	9.83	10.63	10.33	0.36
9	20	8.97	10.09	9.73	0.32
14	4	8.80	9.50	9.25	0.33
20	1	10.00	10.00	10.00	0.00
21	3	9.77	10.03	9.88	0.13
22	8	9.81	10.21	9.99	0.12
23	18	9.46	9.90	9.65	0.12
Means	8	9.25	10.33	9.77	0.36

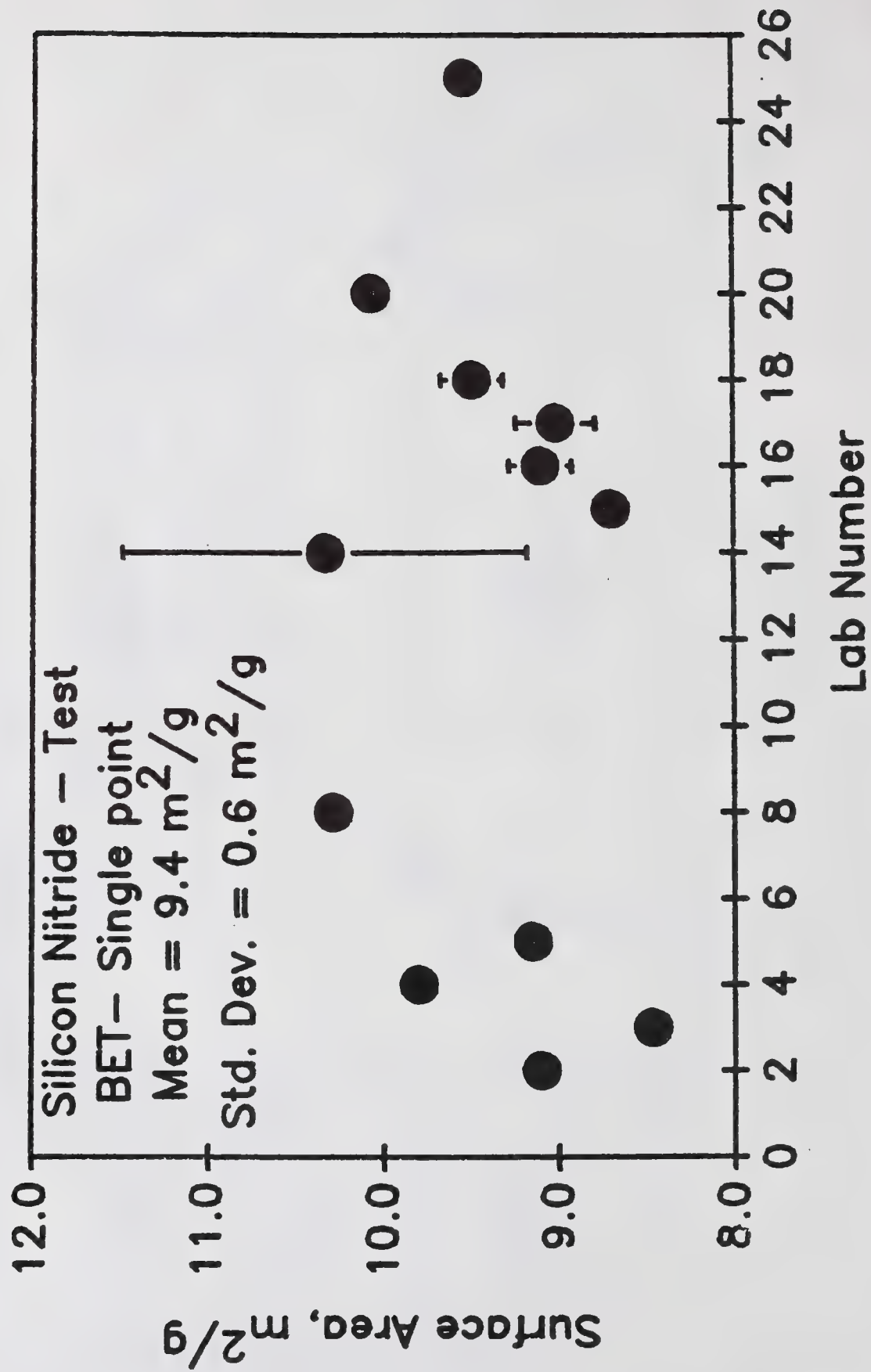


Figure 13. Specific surface area of SNT powder as reported by participating labs using single point BET method.

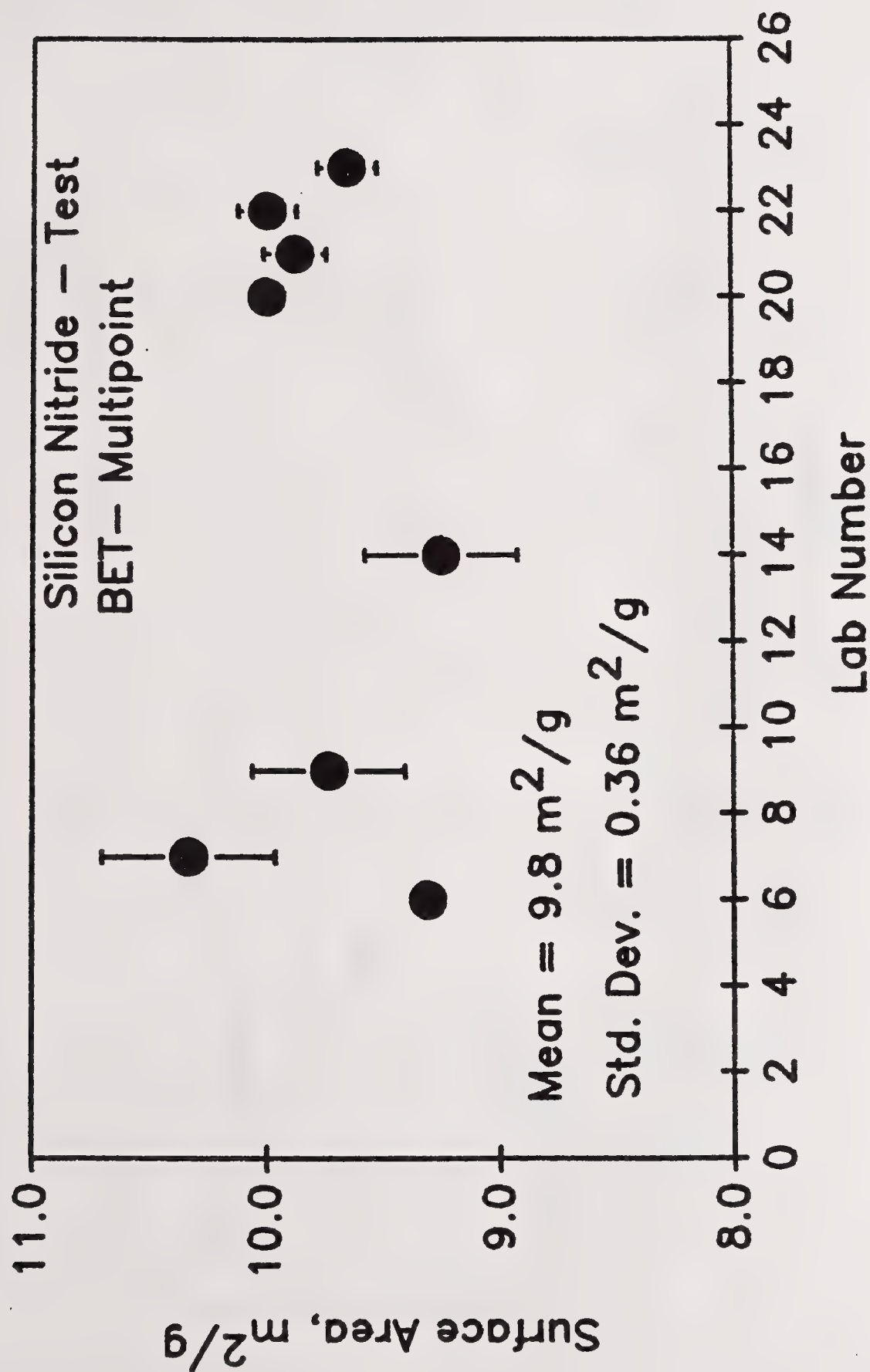


Figure 14. Specific surface area of SNT powder as reported by participating labs using multipoint BET method.

3.2.3. Silicon Carbide (SiC) Powder

Table 3.2.3.1 Single-Point Determination

Material: Silicon Carbide
Property: SINGLEPT
Method: 1

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	4	12.36	12.53	12.44	0.08
2	1	12.80	12.80	12.80	0.00
3	12	12.30	12.90	12.71	0.19
4	12	13.63	13.79	13.72	0.05
8	6	15.41	16.79	16.05	0.66
15	1	11.92	11.92	11.92	0.00
16	3	12.60	12.90	12.70	0.17
17	33	12.94	13.20	13.04	0.07
18	9	12.36	14.05	13.37	0.52
20	1	14.31	14.31	14.31	0.00
25	1	13.70	13.70	13.70	0.00
Means	11	11.92	16.05	13.34	1.12

Table 3.2.3.2 Multi-point Determination

Material: Silicon Carbide
Property: MULTIPT
Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	4	13.58	13.85	13.71	0.11
6	1	11.67	11.67	11.67	0.00
7	3	15.92	16.38	16.20	0.24
9	9	14.42	14.83	14.63	0.13
10	3	15.02	15.43	15.19	0.21
13	5	13.26	13.84	13.58	0.22
21	3	14.68	14.94	14.77	0.14
22	4	14.93	15.67	15.27	0.36
23	12	13.80	14.51	14.27	0.18
Means	9	11.67	16.20	14.36	1.29

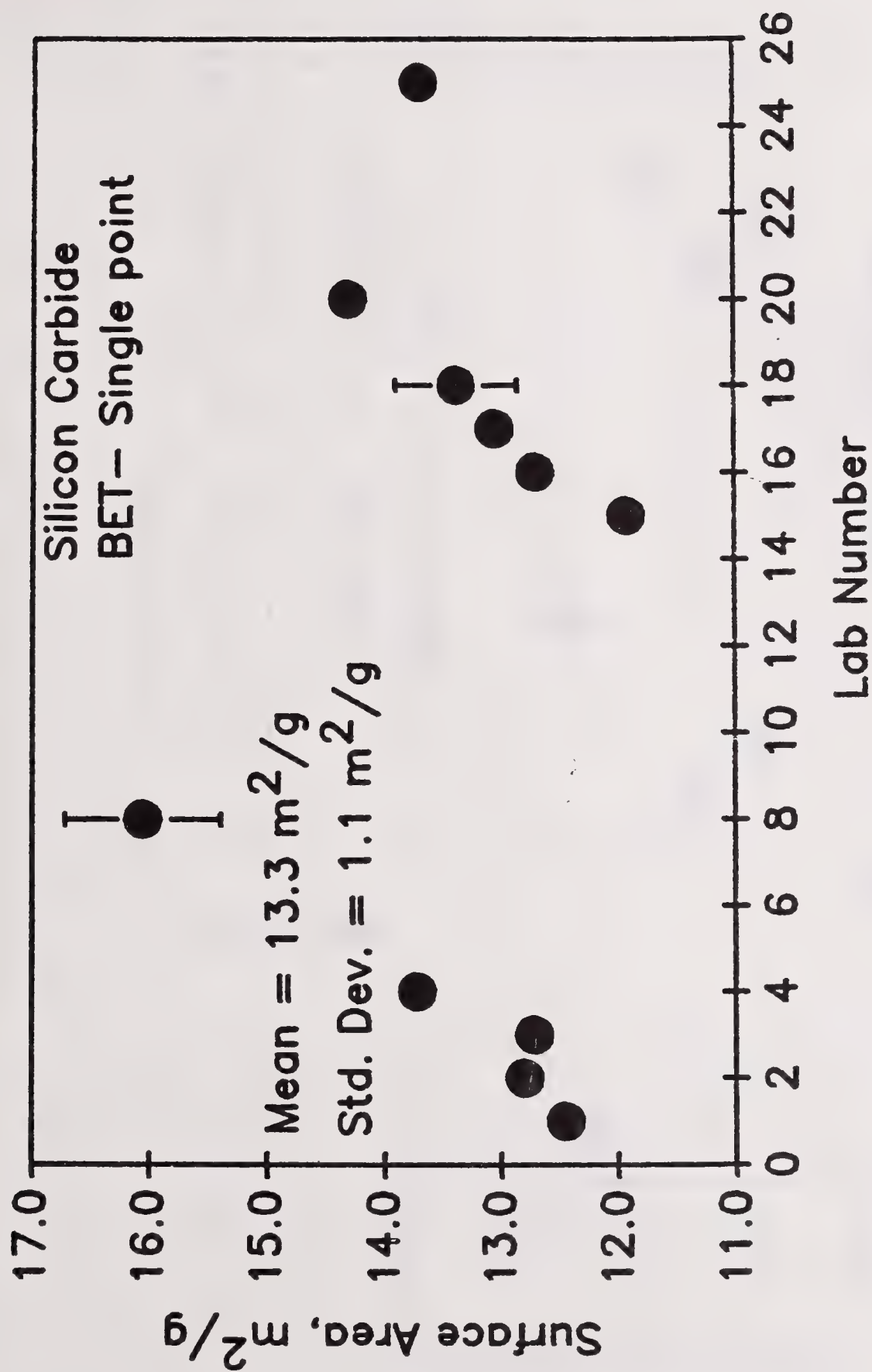


Figure 15. Specific surface area of SiC powder as reported by participating labs using single point BET method.

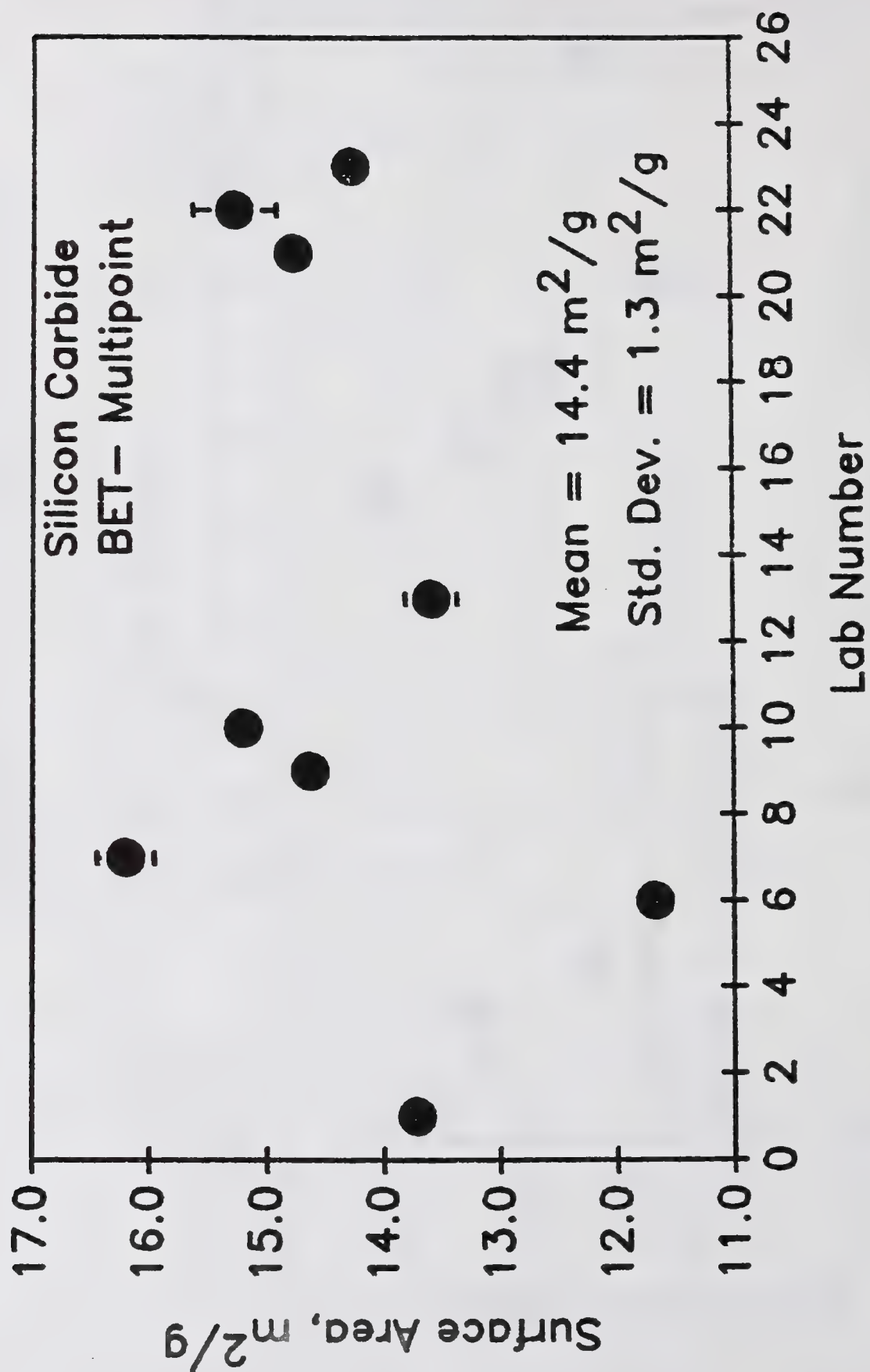


Figure 16. Specific surface area of SiC powder as reported by participating labs using multipoint BET method.

3.2.4. Silicon (Si) Powder

Table 3.2.4.1 Single-Point Determination

Material: Silicon
Property: SINGLEPT
Method: 1

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	4	1.63	1.88	1.78	0.10
2	1	1.80	1.80	1.80	0.00
3	12	1.80	2.00	1.88	0.07
4	12	1.79	2.03	1.94	0.10
5	4	1.70	1.90	1.80	0.11
8	6	2.30	2.36	2.32	0.02
15	1	1.50	1.50	1.50	0.00
16	5	1.96	1.99	1.96	0.01
17	34	1.96	2.05	2.00	0.02
18	9	1.84	1.99	1.91	0.05
20	1	2.25	2.25	2.25	0.00
Means	11	1.50	2.32	1.92	0.22

Table 3.2.4.2 Multi-point Determination

Material: Silicon
Property: MULTIPT
Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	4	1.78	2.02	1.93	0.10
6	1	1.71	1.71	1.71	0.00
9	9	2.16	2.61	2.27	0.17
22	8	1.95	5.32	3.32	1.48
23	12	1.78	2.06	1.97	0.07
Means	5	1.71	3.32	2.24	0.63

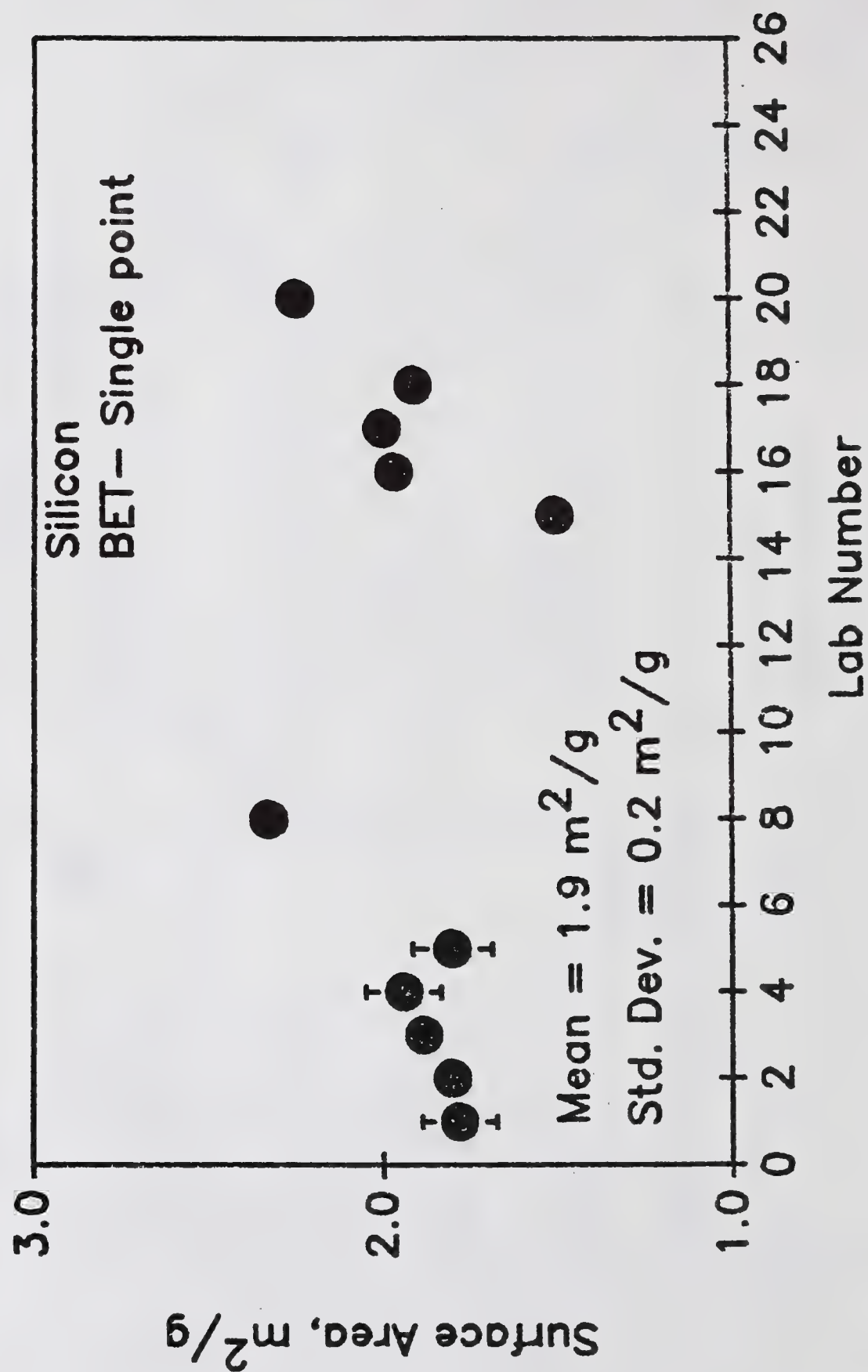


Figure 17. Specific surface area of Si powder as reported by participating labs using single point BET method.

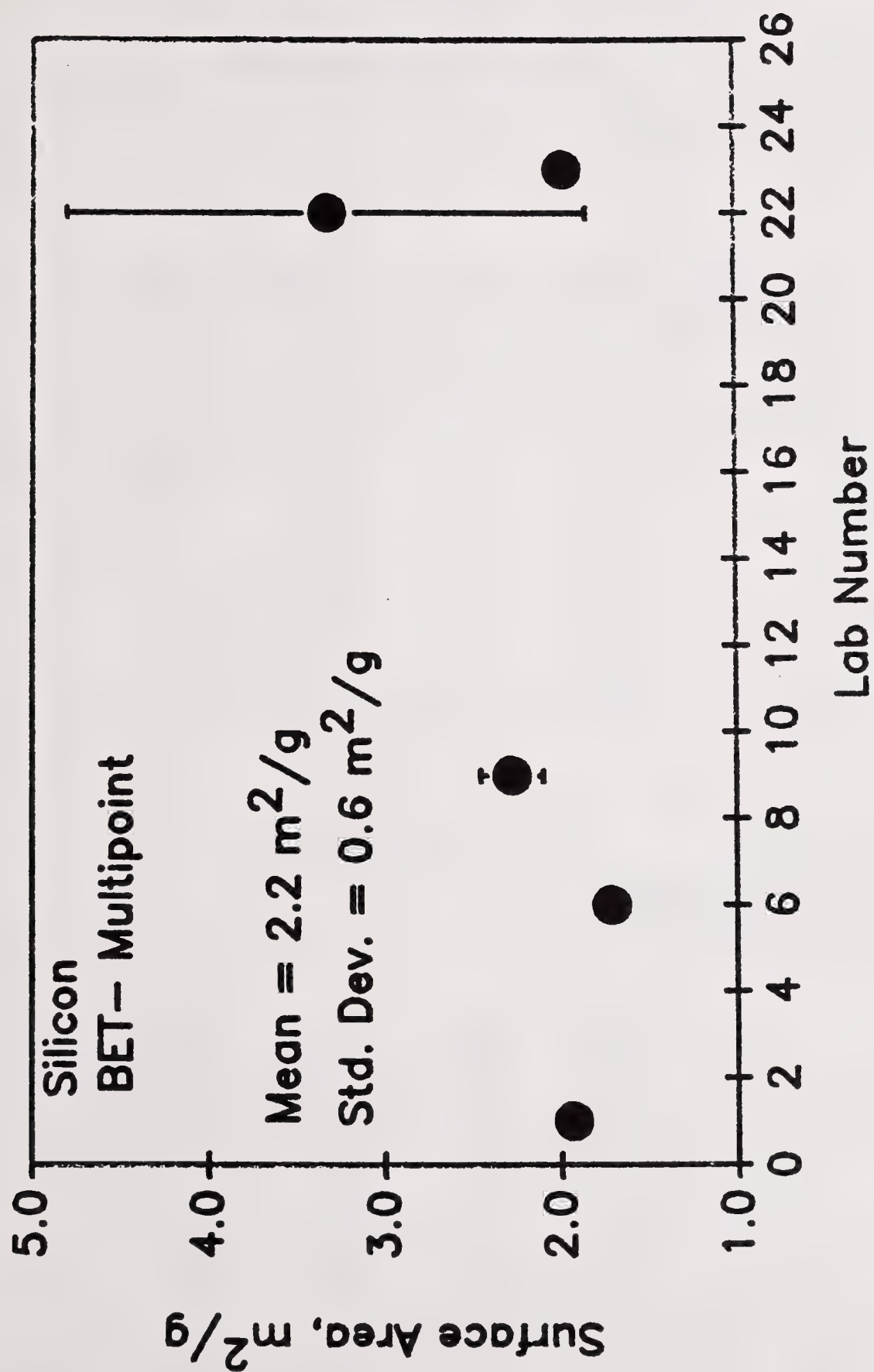


Figure 18. Specific surface area of Si powder as reported by participating labs using multipoint BET method.

3.2.5. Yttria-Zirconia (YSZ) Powder

Table 3.2.5.1 Single-Point Determination

Material: Zirconia
Property: SINGLEPT
Method: 1

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	2	16.93	17.04	16.98	0.07
2	2	15.40	15.70	15.55	0.21
4	24	18.70	19.50	19.05	0.24
6	1	17.12	17.12	17.12	0.00
11	2	14.10	16.30	15.20	1.55
14	3	16.10	17.00	16.53	0.45
15	1	15.80	15.80	15.80	0.00
16	12	17.20	19.00	18.08	0.68
17	18	16.05	17.05	16.40	0.33
18	17	17.60	19.80	18.95	0.63
20	3	16.90	17.16	17.05	0.13
24	3	15.30	16.50	15.80	0.62
Means	12	15.20	19.05	16.87	1.27

Table 3.2.5.2 Multi-Point Determination

Material: Zirconia
Property: MULTIPT
Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	2	17.53	17.67	17.60	0.09
6	1	17.62	17.62	17.62	0.00
8	3	18.61	19.09	18.78	0.26
9	3	18.91	18.99	18.96	0.04
14	2	18.20	18.40	18.30	0.14
19	9	18.03	18.45	18.23	0.15
22	11	18.30	19.90	19.01	0.60
Means	7	17.60	19.01	18.35	0.59

3.3. STATISTICAL ANALYSIS, R. Munro, NIST

The Brunauer-Emmett-Teller (BET) gas adsorption measurement technique was used by the participants to measure the specific surface areas of the powders. There were two primary implementations of the method, denoted single point BET and multi-point BET. In the single point BET procedure, the measurement was conducted at the saturation vapor pressure of the liquified adsorbent gas. In the multi-point BET procedure, measurements were conducted at three or more adsorption pressures.

In both procedures, specimen preparation can affect the BET measurement significantly. Agglomeration of the particles and residual adsorbed gases, for example, necessarily influence the amount of adsorption that can occur during the test. Thus, sampling procedures, degassing conditions, and the choice of adsorbate gas can all be expected to contribute to variations in the results. This expectation appeared to be confirmed by the measures of precision for both methods, Figures 21-25, which had wide variations among the individual laboratories.

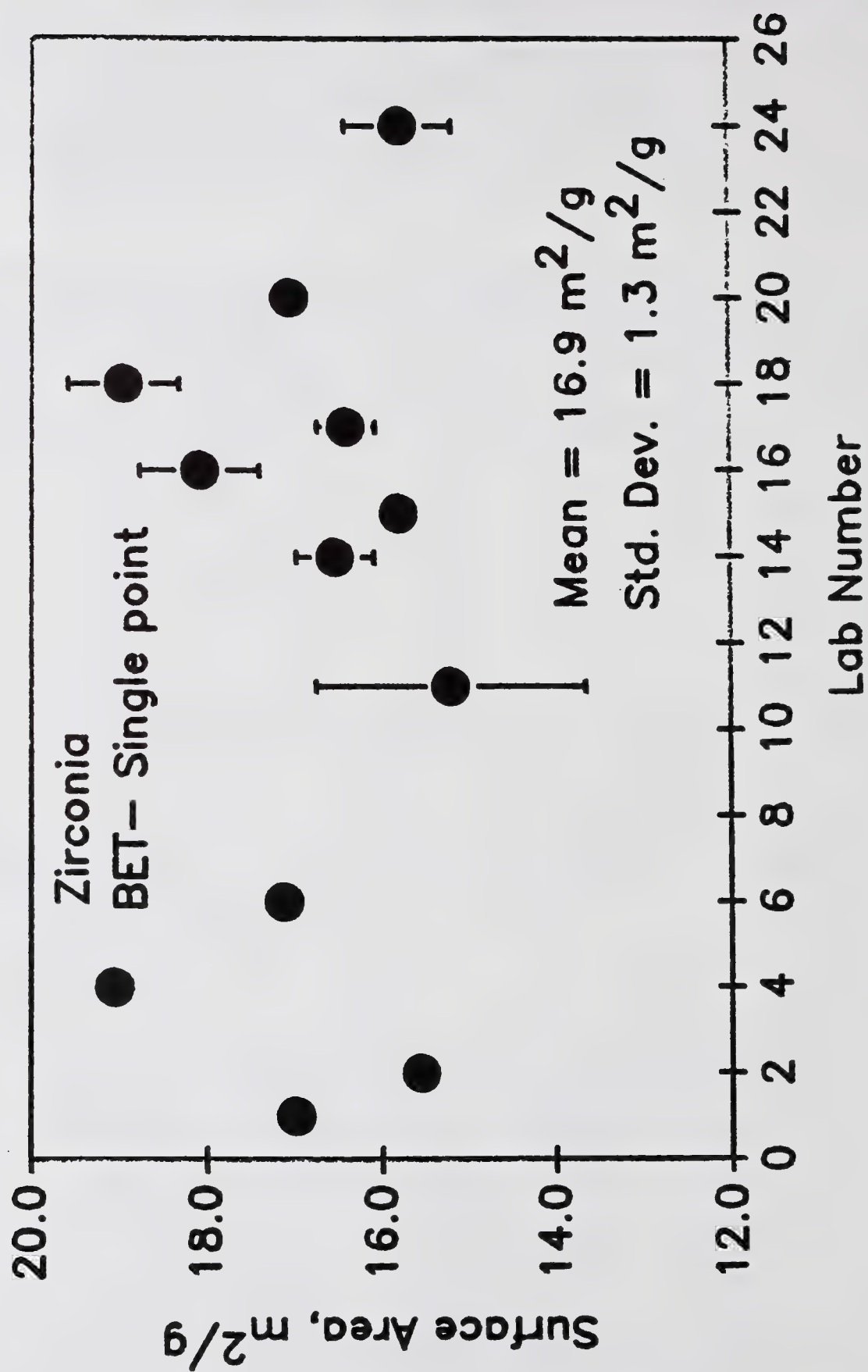


Figure 19. Specific surface area of YSZ powder as reported by participating labs using single point BET method.

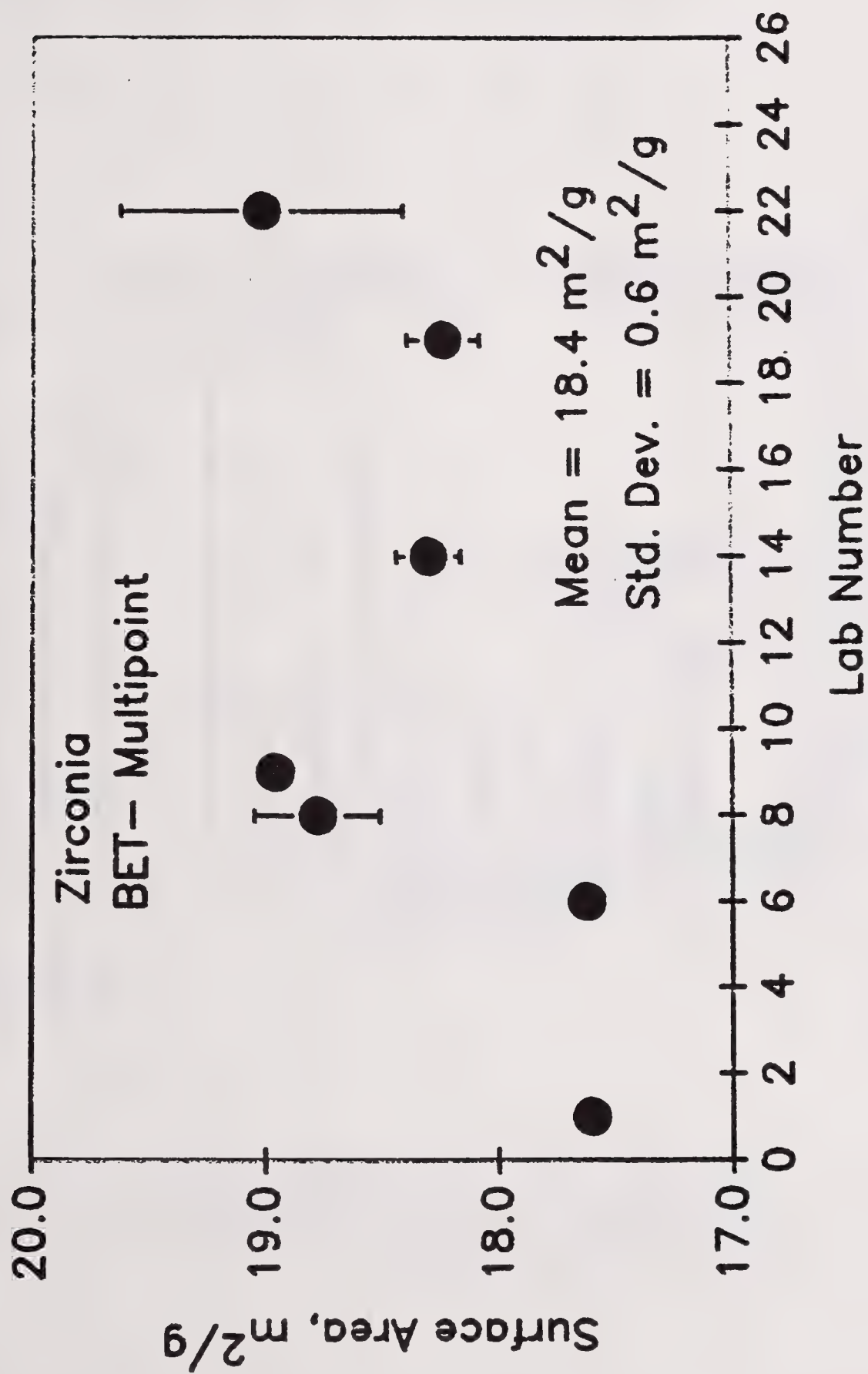


Figure 20. Specific surface area of YSZ powder as reported by participating labs using multipoint BET method.

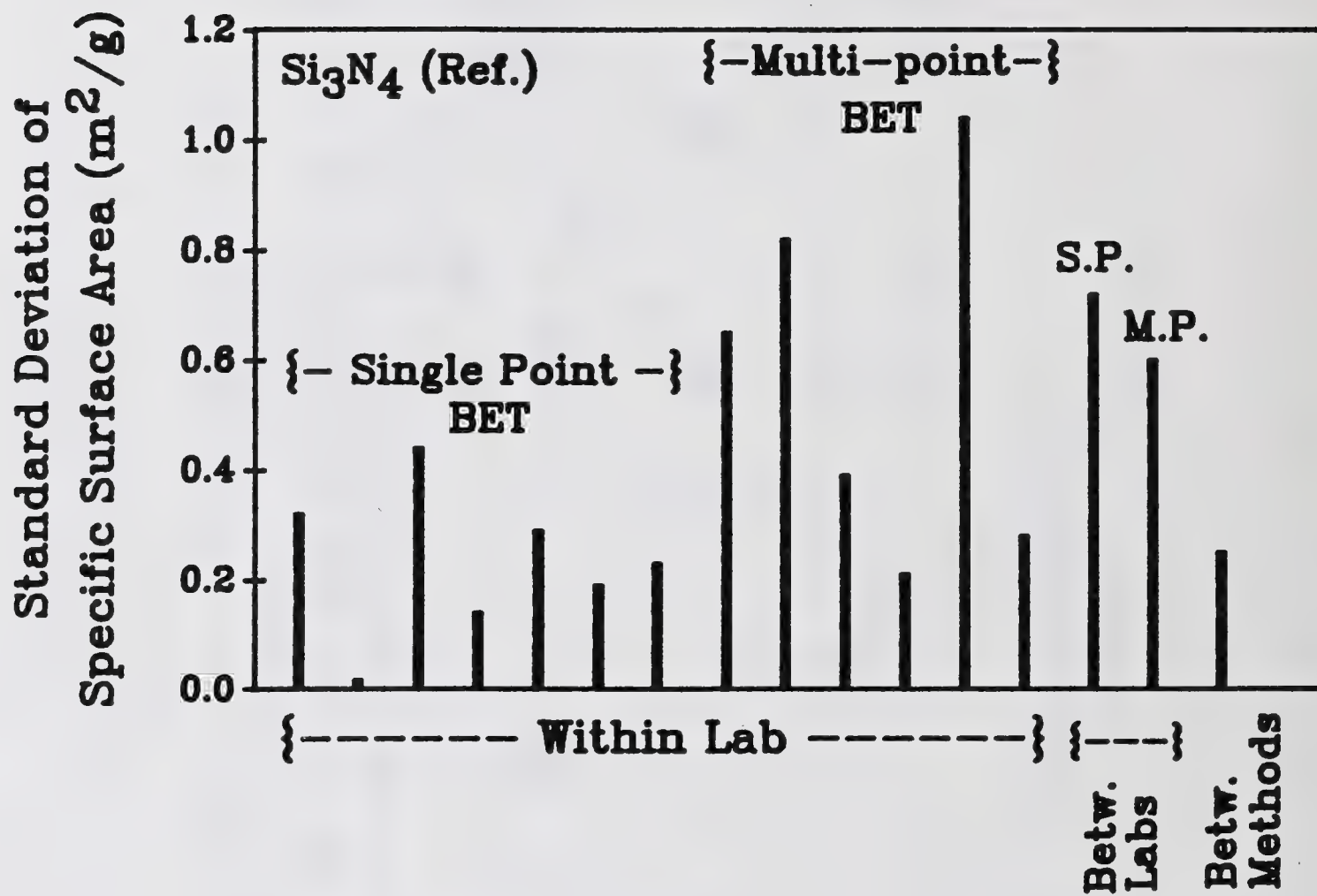


Figure 21.

Within lab, between lab, and between methods standard deviations in the measurement of specific surface area for the Si_3N_4 (Ref.) powder as measured by various laboratories using a variety of methods.

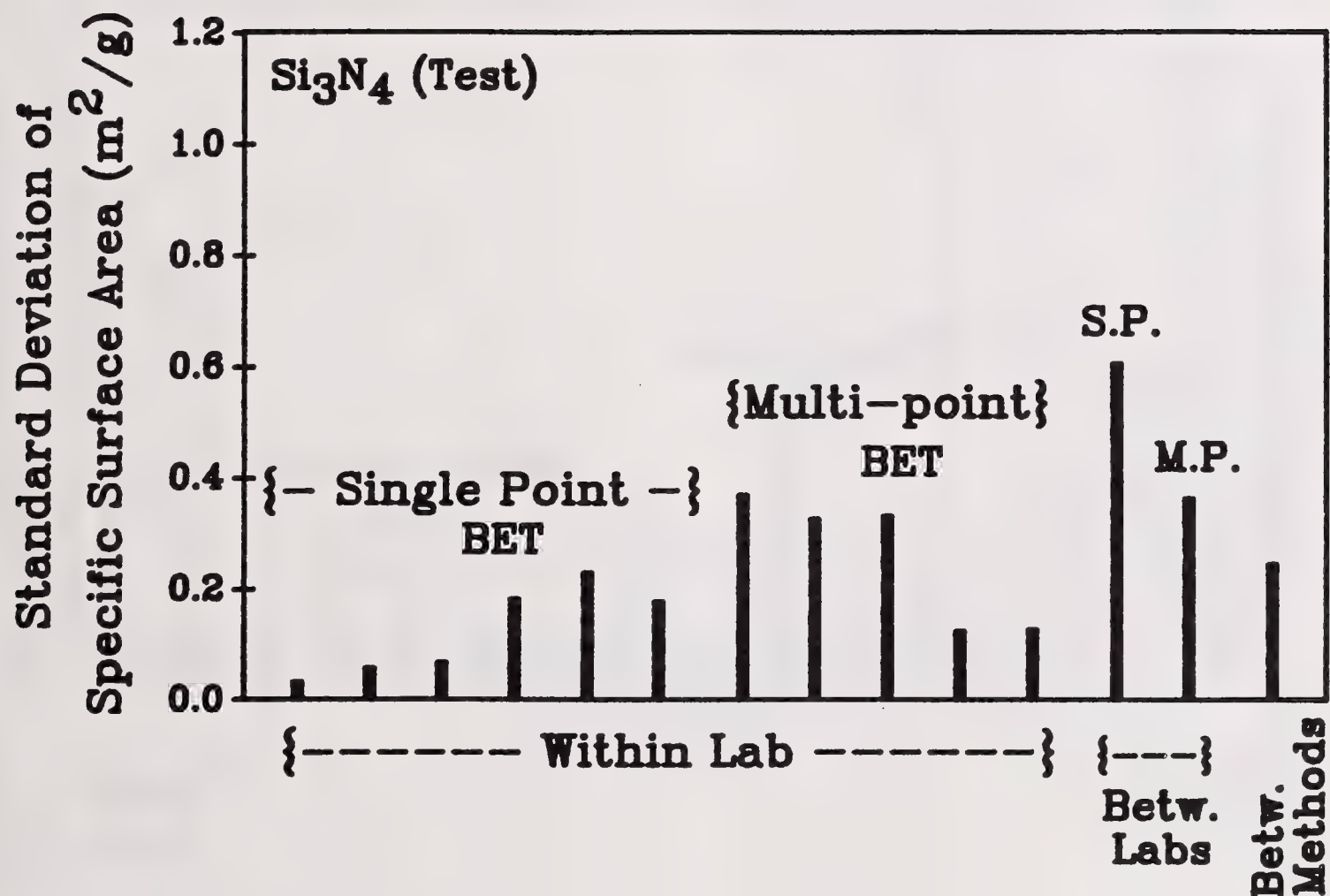


Figure 22.

Within lab, between lab, and between methods standard deviations in the measurement of specific surface area for the Si₃N₄(Test) powder as measured by various laboratories using a variety of methods.

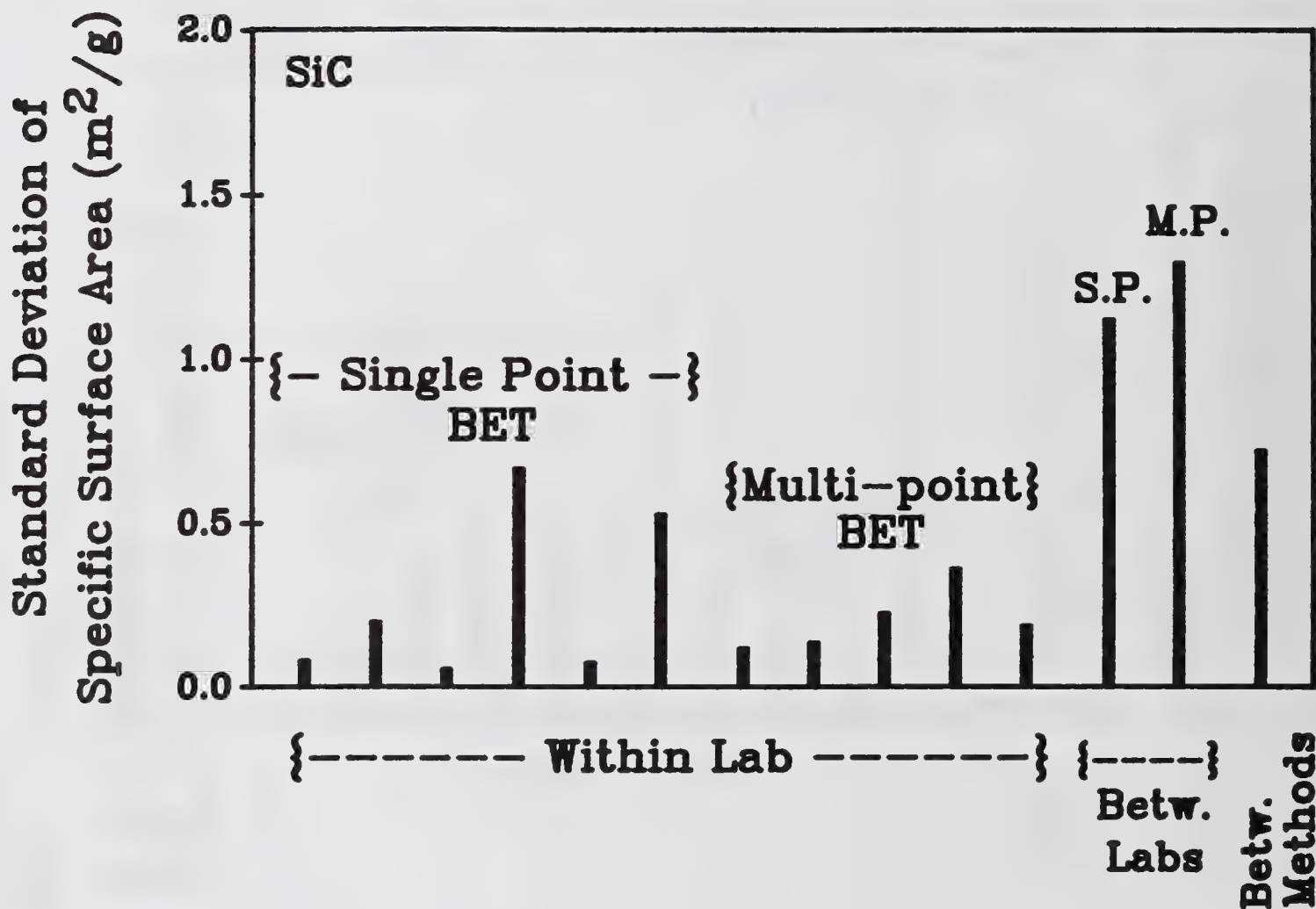


Figure 23.

Within lab, between lab, and between methods standard deviations in the measurement of specific surface area for the SiC powder as measured by various laboratories using a variety of methods.

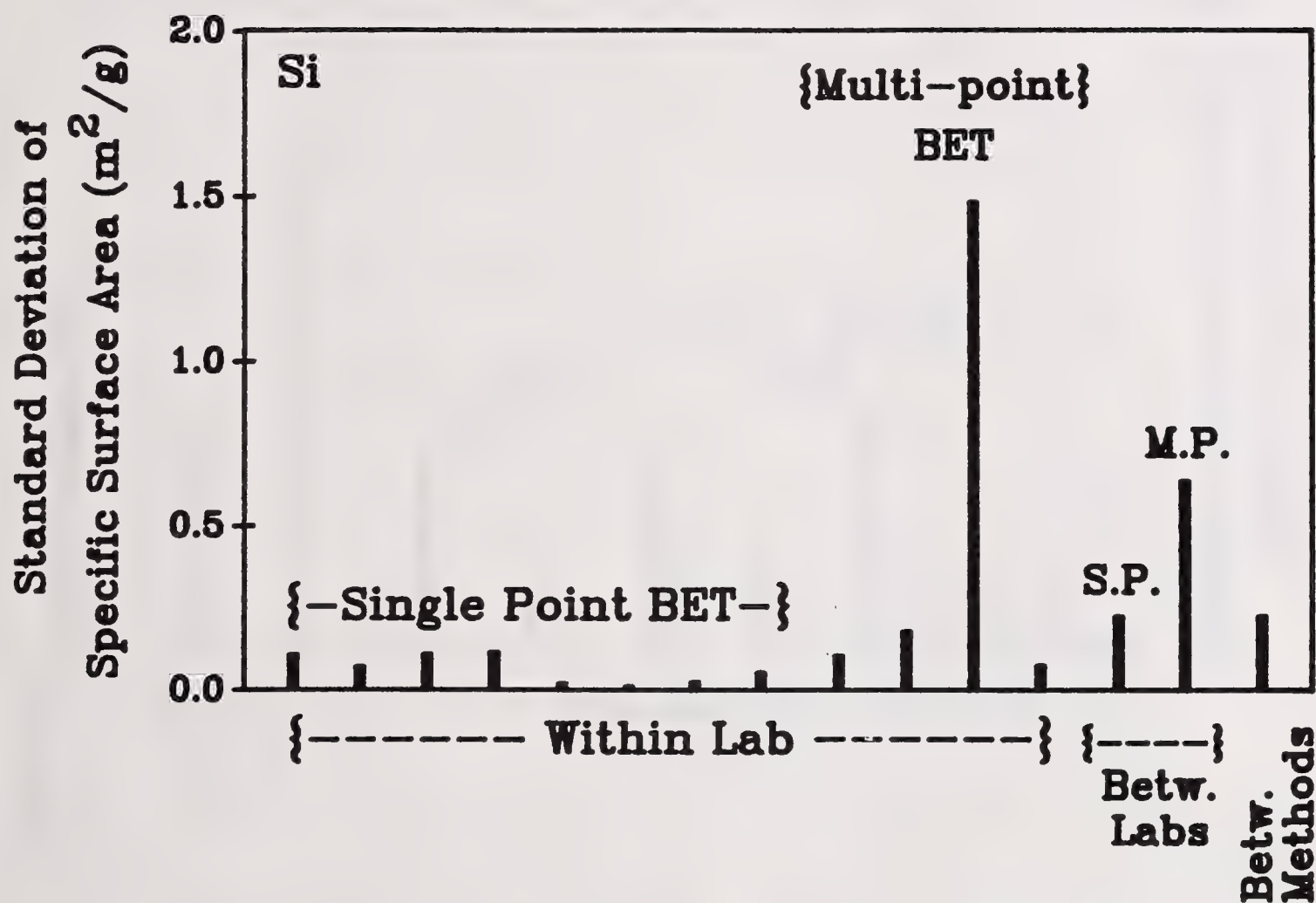


Figure 24.

Within lab, between lab, and between methods standard deviations in the measurement of specific surface area for the Si powder as measured by various laboratories using a variety of methods.

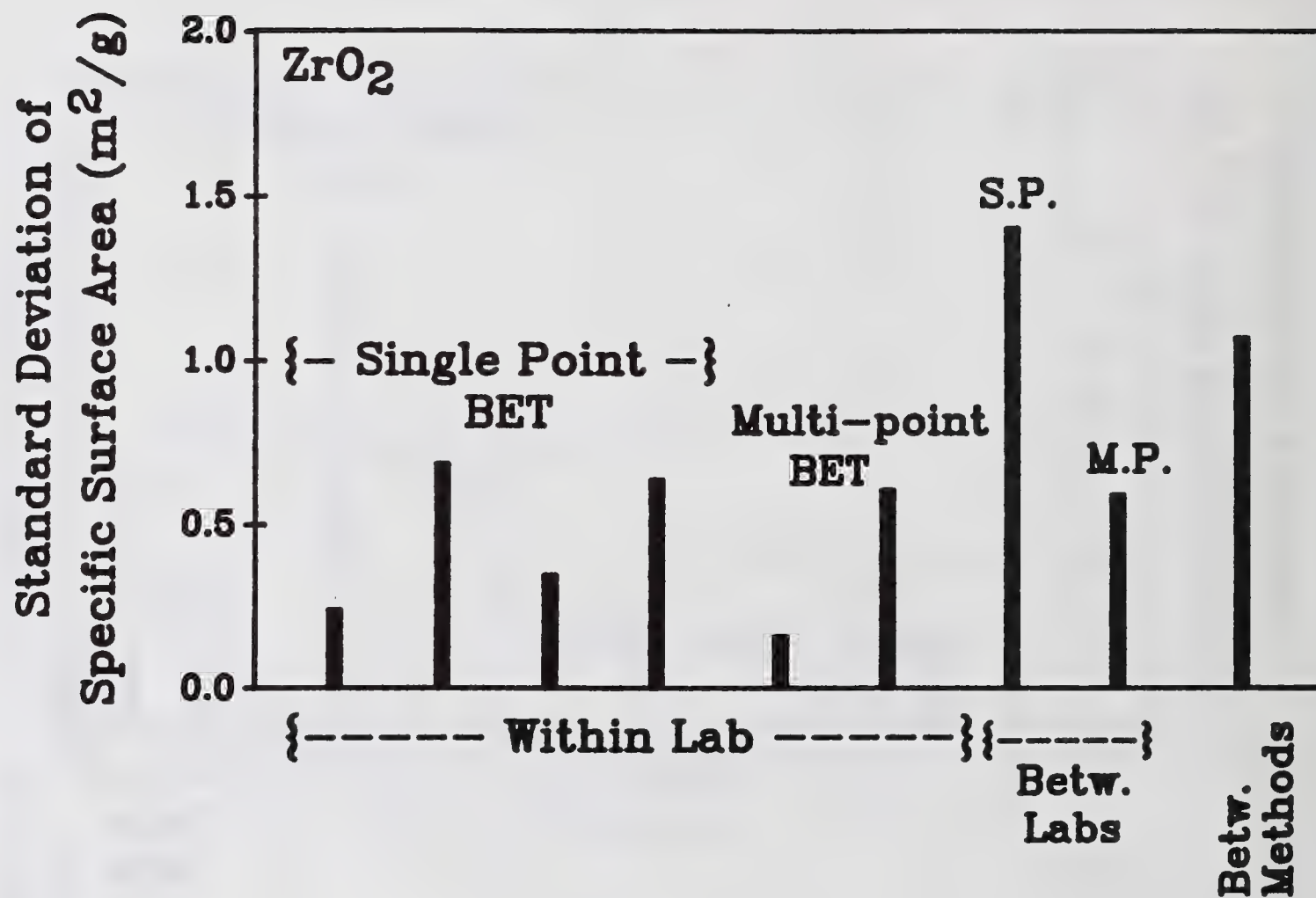


Figure 25.

Within lab, between lab, and between methods standard deviations in the measurement of specific surface area for the ZrO₂ powder as measured by various laboratories using a variety of methods.

4. PARTICLE SIZE AND PARTICLE SIZE DISTRIBUTION (PS & PSD)

H. Hausner, Technische Universität Berlin; S. Malghan, NIST

4.1. MEASUREMENT METHODS

4.1.1. Methods Available

in the case of zirconium oxide, silicon carbide and silicon nitride, the particle size and size distribution has a significant influence on the densification of these powders during sintering. The particle size of silicon is important for its nitridation behavior during silicon nitride powder synthesis from the metal. Properties of the Si_3N_4 powder will directly affect the properties of the final ceramic product in the case of reaction bonded silicon nitride.

in particle size analysis, the method used will depend on the particle size of the powder under investigation. The methods shown in Table 4.1.1.1 can be used for different ranges of particle sizes.

Table 4.1.1.1 Particle Size Range and Analysis Method

Range (μm)	Method
(1) 10000 - 5	Sieving (including micromeshsieves)
(2) 60 - 0.2	Gravitational Sedimentation
(3) 5 - 0.02	Centrifugal Sedimentation
(4) 600 - 0.1	Light Scattering
(5) 3 - 0.003	Photon Correlation Spectroscopy
(6) 800 - 0.5	Electrical Sensing Zone
(7) 5 - 0.02	Scanning Electron Microscopy
(8) 5 - 0.001	Transmission Electron Microscopy

Methods (2) through (4) were used to the greatest extent by the laboratories which participated in this investigation. Method (1) was not used at all and will not be considered further here. Very limited data were reported for methods (5) and (6). In the sections which follow, methods (2) through (4) will be discussed. Method (5) was used only by two laboratories (9 and 23) for measurements on the YSZ powder. Method (6) was used only by Lab 16 for Si powder. Methods (7) and (8) are discussed under Particle Morphology in Chapter 5.0.

Methods for measuring sizes and size distribution of fine powders have been reviewed and evaluated in recent years⁶⁻⁹. In addition, many articles have appeared in the particle characterization literature comparing two techniques, such as light-scattering with the longer established gravitational sedimentation and electrical sensing zone methods.

4.1.2. Gravitational Sedimentation (Sedigraph)

Use and Significance

The Sedigraph particle size analyzer measures the sedimentation rates of particles dispersed in a liquid, calculates the equivalent spherical diameter (ESD) on the basis of Stoke's law of sedimentation, and plots the data as cumulative mass percent versus ESD. Sedigraphs using light or x-ray detection are in use. The instrument using x-ray is described here. The time required to analyze a particle size distribution is significantly decreased by continuously changing the sedimentation cell position so as to decrease the effective sedimentation depth. Nevertheless, the duration of an analysis for a single specimen may be from 1 to 2 hours. Although the analytical range of the instrument is 100 to 0.1 μm , the actual operating range is controlled by the rheological characteristics of the powder in liquid to be analyzed. The accuracy of the instrument decreases for particle sizes below 1 μm due to the increase of diffusion effects in the liquid.

Principle

For an x-ray type Sedigraph, the intensity, I , of the transmitted x-ray beam is proportional to the mass, C , of powder in the beam according to the absorption law

$$I = I_0 \exp(-BC) \quad (2.1)$$

where B is a mass absorption constant. The x-ray density is defined as

$$D = \log_{10}(I/I_0) \quad (2.2)$$

The PSD is calculated on the basis of Stoke's law at intervals of the cell position using the measured settling depth, powder and liquid densities, and liquid viscosity.

Instrument and Experimental Parameters

Size distribution analysis by Sedigraph consists of two steps: (1) sample preparation and (2) size analysis. The parameters influencing sample preparation for particle size analysis are:

- dispersion method
- dispersion liquid
- dispersion agent

- deagglomeration
- deagglomeration time
- time elapsed before analysis
- solid-liquid ratio
- pH of suspension
- temperature of suspension
- volume of sample

Special liquids containing surfactants (e.g., Sedisperse™) are available from instrument manufacturers; however, many common liquids for which the density and viscosity are known over a temperature range around room temperature may be used. A variety of surfactants (dispersion agents) are available. Their use in practice is governed largely by empiricism and art. Deagglomeration is carried out by use of some form of shear and impact energy - e.g., ultrasonic bath or probe, blender, or stirrer. It is essential that the means used to deagglomerate the powder produce disruption of all mechanically bound agglomerates and produce a uniform suspension of all the particles. The dispersed suspension is placed in the instrument compartment and pumped into the sample cell.

As can be seen from the compilation of experimental conditions given in Table 4.1.2.1, the experimental conditions for sample preparation differed significantly from laboratory to laboratory.

Sample density, liquid density and liquid viscosity are used to calculate a run parameter to which the instrument is set, or they are entered into a run file if the instrument is computer controlled.

The x-ray Sedigraph uses a finely collimated beam of low-energy xrays to obtain a direct measure of mass concentration at a series of accurately determined levels in a sample cell. A scintillation detector monitors the beam which is transmitted through the cell. With the use of the internal electronics a continuous display of the particle size as ESD versus cumulative mass percent is produced by an x-y plotter. Output of the instrument-response permits further treatment of the data to be carried out, such as a plot of ESD versus mass frequency.

Calibration and Standards

The instrument is calibrated using either manufacturer provided standard powders or those obtained from organizations as the National Institute of Standards and Technology and the Community Bureau of References (BCR). At present, no certified standard ceramic powders, especially in the range 0.1-1.0 μm , are available for calibration purposes.

The following ASTM procedure may serve as a guide in the use of the Sedigraph for ceramic powders:

Table 4.1.2.1 Methods Used for Dispersion of Powders in Gravitation Sedimentation

<u>Powder Type</u>	<u>Media</u>	<u>Dispersant</u>	<u>Dispersion Method</u>	<u>Labs. Using Procedure, #</u>
1. Silicon Nitride, R&T	Sedisperse A-11	Mineral Spirit-Based Liquid, Micromeritics, Inc.	Not Specified	4, 15
2. Silicon Nitride, R&T	Sedisperse A-11	Mineral Spirit-Based Liquid, Micromeritics, Inc.	Ultrasonic probe for 1-5 min	1, 3, 6
3. Silicon Nitride, R&T	Sedisperse A-11	Mineral Spirit-Based Liquid, Micromeritics, Inc.	Ultrasonic bath	17, 23
4. Silicon Nitride, R	Water	Lignosulfonate	Ultrasonic bath for 30 min	4
5. Silicon Nitride, R&T	Water, pH 10	Lignosulfonate	Ultrasonic bath for 30 min	16
6. Silicon Nitride, R&T	Water, pH 10	Lignosulfonate	Ultrasonic bath for 30 min	9, 18
7. Silicon Carbide, Silicon	Sedisperse A-11	Mineral Spirit-Based Liquid	Ultrasonic probe for 1-5 min	1, 3, 6
8. Silicon Carbide, Silicon	Sedisperse A-11	Mineral Spirit-Based Liquid	Ultrasonic bath 30, 60 min	23
9. Silicon Carbide, Silicon	Sedisperse A-11	Mineral Spirit-Based Liquid	Not Specified	4
10. Silicon Carbide	Water, pH 10	Lignosulfonate + wetting agent	Ultrasonic probe for 10 min	16
11. Silicon Carbide	Water, pH 9	Sodium hydroxide	Ultrasonic probe for 3 min	9
12. Silicon Carbide, Silicon	Water	Butylamine	Not Specified	15
13. Silicon Carbide	Water	Sodium pyrophosphate	Not Specified	10

14. Silicon	Sedisperse A-11	Mineral Spirit- Based Liquid, Micromeritics, Inc.	Ultrasonic bath for for 20, 40, 60 min	17
15. Silicon	Isopropyl alcohol	Ammonium hydroxide	Ultrasonic probe for 3 min	9
16. Silicon	Water	Napcosant	Ultrasonic probe for 2 min	18
17. Silicon	Water	Lignosulfonate	Ultrasonic probe for 10 min	16
18. Zirconia	Water	0.05% sodiumpyro- phosphate	Ultrasonic bath for 2 min	6
19. Zirconia	Sedisperse A-13-583	Mineral Spirit- Based Liquid, Micromeritics, Inc.	Ultrasonicated for 10 min	17
20. Zirconia	Sedisperse A-11	Mineral Spirit- Based Liquid, Micromeritics, Inc.	Ultrasonicated for 30 min	23
21. Zirconia	Water	Dispex N-40	Ultrasonicated for 2 min	18
22. Zirconia	Water, pH 10	Lignosulfonate	Ultrasonicated with a probe for 10 min	16

ASTM C958-81. Standard method for particle size distribution of alumina or quartz by x-ray monitoring of gravity sedimentation.

Nomenclature and theoretical methods for representing and analyzing particle size distributions are recommended in

DIN 66141. Darstellung von Korn-(Teilchen)großenverteilungen; Grundlagen
[Representation of particle size distribution; basic standards]

These recommended representations are relevant to all of the methods considered here.

4.1.3 Centrifugal Sedimentation

Use and Significance

Since diffusional-remixing significantly interferes with gravitational sedimentation at particle sizes less than $1\text{ }\mu\text{m}$, the use of centrifugal forces is preferred for particle sizes in the range of 0.1 to $2\text{ }\mu\text{m}$. Sedimentation in a centrifugal field is subject to the same limitations of concentration of the solid phase, particle shape, and electroviscosity effects as in gravity sedimentation. In addition, analysis of centrifugal data is complicated by the variation of the centrifugal force on a particle as a function of distance.

As the range of particle size to be determined decreases from 0.1 to $0.01\text{ }\mu\text{m}$, a large centrifugal force becomes essential to achieve adequate sedimentation rates. In such cases, a centrifuge which can produce accelerations between $200,000\text{ g}$ and $400,000\text{ g}$ ($g = 9.80\text{ m/s}^2$), in the vicinity of $60,000\text{ rpm}$, may be required. At these speeds and particle sizes, the effect of Brownian motion (i.e., diffusion) may still have an influence on sedimentation rates.

Principle

Two instrumental methods have been commercialized in recent years: (1) the line-start method and (2) the homogeneous suspension method. Brief descriptions of these two methods follow. Only instruments of the homogeneous suspension type were used in this study.

In the typical line-start instrument, a thin layer of host liquid is spread over a rapidly spinning disk; then a small amount of suspension is injected as a thin inner annular ring. Suspended particles are detected at an outer point in the host liquid by a photodiode with the heavier particles travelling past the detection point before the lighter particles. Mathematical extraction of the particle size distribution data from the intensity time data is not greatly encumbered by approximations to integral solutions. However, significant experimental errors may be encountered for large particle sizes ($> 2\text{ }\mu\text{m}$) since these particles may be swept out of the suspension rapidly for an accurate resolution of the size distribution. Experimentally, errors may arise from hydrodynamic streaming of the suspension through the host liquid.

In the typical homogeneous suspension instrument, a cell containing a homogeneous suspension of the sample is placed with the axis of the cell along a radial direction of a high-speed centrifuge. The centrifuge is accelerated quickly or in a programmed manner to a stable rotational velocity and the particle density at one location in the cell is measured as a function of time with a photodiode. Since this method measures the light absorption by the total time-dependent population of particles in a small detection volume, mathematical analysis of the time intensity data must treat the total time-dependent particle size distribution. This requires the use of approximations of an integral equation. The algorithms used by instrument manufacturers are usually proprietary so that these instruments typically perform as a "black box." In application, homogeneous suspension instruments are usually somewhat easier and convenient to use than are the line-scan instruments. In addition, the homogeneous suspension instruments usually provide for determination of sedimentation of large sizes ($> 1 \mu\text{m}$) by a gravitational settling determination.

Instrument and Experimental Parameters

A single method cannot be described which is applicable to all instruments; however, a general method is described here which may be more-or-less typical for homogeneous suspension instruments. The powder which is dispersed in an appropriate liquid medium containing a dispersant is transferred into a sedimentation cell. A reference cell filled with a clear sample of the dispersant liquid (blank) is used to balance the centrifuge mechanically and to provide an optical reference. The centrifuge is typically spun at speeds up to 10,000 rpm, producing centrifugal force up to 9000 g. This may permit particle sizes down to $0.01 \mu\text{m}$ to be determined; the lower limit depends upon particle density. The particle size is calculated from the change in optical absorbance of the suspension as a function of time. Instruments may have a built-in correction for absorbance coefficient which minimizes any measurement error caused by differences in light attenuation efficiencies of particles with different diameters. A large number of different data presentation systems and formats are available with these instruments.

Sample preparation considerations are similar to those outlined in Section 4.1.2., above. The experimental conditions used in this study are summarized in Table 4.1.3.1.

Calibration and Standards

No specific standards based on ceramic powders are available for centrifugal instruments. A standard procedure was developed by ASTM for centrifugal sedimentation, but the procedure was based on instrumentation which is no longer commercially available. However, this procedure may be consulted for guidance:

ASTM C678-75 (Reapproved 1985). "Standard Method for Particle Size Distribution of Alumina or Quartz Using Centrifugal Sedimentation."

Table 4.1.3.1 Methods Used for Dispersion of Powders in Centrifugal Sedimentation

<u>Powder Type</u>	<u>Media</u>	<u>Dispersant</u>	<u>Deagglomeration Method</u>	<u>Labs. Using This Procedure, #</u>
1. Silicon Nitride, R&T	Sedisperse A-11	Mineral Spirit Based Liquid, Micromeritics, Inc.	Ultrasonic probe for 3 min	1
2. Silicon Nitride, R & Silicon	Water	Micro A87-578	Not Specified	13
3. Silicon Nitride, R	Water	Sodium pyrophosphate	Ultrasonicated for 6-12 min	11, 14
4. Silicon Nitride, R	Water	CMS-70	Not Specified	19
5. Silicon Nitride, T	Water	Sodium pyrophosphate	Ultrasonicated for 3, 12, 30 min	14
6. Silicon Nitride, T	Water	Sodium pyrophosphate	Ultrasonicated in a bath for 5 min	5
7. Silicon Nitride, T	Water	CMS-70	Not Specified	19
8. Silicon Carbide, Silicon	Sedisperse A-11	Mineral Spirit Based Liquid, Micromeritics, Inc.	Not Specified	1
9. Silicon Carbide, Silicon	Not Specified	Not Specified	Not Specified	2
10. Silicon	Ethylene alcohol	Not Specified	Not Specified	19
11. Zirconia	Sedisperse A-12	Not Specified	Not Specified	1
12. Zirconia	Not Specified A-12	50% Glycerol	Not Specified	13
13. Zirconia	Water	Sodium pyrophosphate	Ultrasonic (200i)	11

4.1.4. Light Scattering

Use and Significance

Of the new methods of particle size distribution analysis, light scattering has grown the most rapidly. The application of light scattering has been increased and refined by improvements in laser technology; computers, especially microcomputers; and computer-based algorithms for data analysis. In light scattering instruments, laser illumination is used. The scattered light data are processed by a combination of mechanical, optical and electronic means. For larger particle sizes ($D \gg \lambda$; where D is the particle size and λ is the wavelength of light) Fraunhofer diffraction theory is used; for smaller sizes (typically $D < 2$ and $\lambda = 0.6 \mu\text{m}$) Lorenz-Mie theory forms the basis for data analysis.

Principle

The form of the scattering pattern for light scattered out of a well collimated beam of light by particles depends upon the dimensions of the particles and the wavelength of light. The relative intensity and spatial distribution of the scattered light is well described by diffraction and scattering theories. Accurate measurement of intensity and spatial distributions enable particle size distributions to be determined. Detailed discussions of Fraunhofer and Lorenz-Mie theories and methods based on them can be found elsewhere⁹.

Instrument and Experimental Parameters

In a typical optical sizer a laser beam from, e.g., a He-Ne laser source, is focused into the sizer's detection zone or is expanded and passed through a glass sample cell in a parallel fashion. A suspension of particles which is stirred and ultrasonicated in a separate chamber is circulated continuously by a peristaltic pump from the chamber and through the sample cell.

The unscattered portion of the incident beam is cut off by a mask since its intensity is so strong that it would swamp the photodetector. The scattered light is passed through a rotating disc which contains apertures arranged in a spiral configuration. A multi-element array detector is used in the optical focal plane to record the light distribution. The digital output of the photodetectors is processed by a computer.

See Section 4.1.2 above for an outline of sample preparation factors. Experimental conditions used in this study are summarized in Table 4.1.4.1.

Calibration and Standards

Light scattering instruments commonly provide data in the form of number density versus particle size, which can be transformed to provide data in terms of mass density versus particle size. Calibration with standards such as latex spheres is routinely practiced.

Table 4.1.4.1 Methods Used for Dispersion of Powders in the Light Scattering Analysis.

<u>Powder Type</u>	<u>Media</u>	<u>Dispersant</u>	<u>Deagglomeration Method</u>	<u>Labs. Using This Procedure, #</u>
1. Silicon Nitride, R&T	Water, pH 10	—	Ultrasonicated with a probe for 3 min	9
2. Silicon Nitride, R&T	Water	Colloid 226035	Ultrasonicated for 1 min	20
3. Silicon Nitride, R&T Silicon	Water, pH 10	Aerosol OT	Ultrasonicated with a probe for 1 min	22
4. Silicon Nitride, R&T	Water	Sodium pyrophosphate	Ultrasonicated for 3 & 12 min	14
5. Silicon Nitride, R&T	Water	Sodium pyrophosphate	Ultrasonicated for 2-3 min	24
6. Silicon Nitride, R	Water	Sodium pyrophosphate	Ultrasonicated in a bath for 4 min	5
7. Silicon Nitride, R	Water	Sodium pyrophosphate	Ultrasonicated for 1 min	21
8. Silicon Nitride, R&T Silicon	Isopropyl alcohol	FVPK-15, polyvinylpyrrolidone	Ultrasonicated for 2 min with a probe	8
9. Silicon Nitride, R&T Silicon	Not Specified	—	Not Specified	7
10. Silicon Nitride, R&T Silicon	Not Specified	—	Ultrasonicated with a probe for 30 min	25
11. Silicon	Water	Tetramethylammonium hydroxide	Ultrasonicated for 1 min	21
12. Silicon	Water	Sodium pyrophosphate	Ultrasonicated in a bath for 10 min	10
13. Zirconia	Water	Sodium pyrophosphate	Ultrasonicated	14
14. Zirconia	Water	Darvan 811 (ml of 1% solution in 9 ml water)	Ultrasonicated with a probe (165W) for 10 min	22
15. Zirconia	Water	Darvan 811, 1% solution	Ultrasonicated for 3 min with probe	9

The following ASTM procedure may be consulted for guidance in the practice of laser light scattering:

ASTM C1070-86. "Determining Particle Size Distribution of Alumina or Quartz by Laser Light Scattering."

4.4.1.5. Results and Discussion

A comparison of the results using statistical analysis is not appropriate since in most cases not enough data are available. Therefore, the discussion will focus on specific examples which were selected to demonstrate the influence of different procedures which were applied for sample preparation. In order to give an overview of the overall scatter of the data, the maximum and minimum D_{50} , D_{90} and D_{10} values obtained with different instruments are shown in Table 4.1.5.1 for the SNR powder independent of the dispersion method. Nearly every laboratory used a different procedure for sample preparation, see Section 4.1.2., and little information was made available, for example, on the ultrasonic power for deagglomeration or on the experimental set-up which was used to transfer the ultrasound to the powder suspension.

Table 4.1.5.1 Particle Size Distribution for the SNR Powder for Different Instrumental Methods

Method		D ₅₀	D ₉₀	D ₁₀
Gravitational Sedimentation	Min.	0.6	1.4	0.2
	Max.	1.4	7.3	0.7

Centrifugal Sedimentation	Min.	1.0	n.a.	n.a.
	Max.	1.7	n.a.	n.a.

Light Scattering	(1) Min.	0.7	2.1	0.3
	Max.	1.5	10.2	0.4
	(2) Min.	0.4	2.0	0.3
	Max.	0.9	9.6	0.3

(1) Microtrac Instrument

(2) Cilas Instrument

In Table 4.1.5.2, the ratio D_{max}/D_{min} is indicated based on the data from Table 4.1.5.1. It can be seen that the ratio is the largest for the D₉₀ values indicating that the influence of the dispersion method had the greatest influence on the large particles or agglomerates.

In the following discussion specific examples will be given to demonstrate the influence of various factors which are mainly responsible for the scatter in the results.

Table 4.1.5.2 Dmax/Dmin Ratio for SNR Powder and Different Instrumental Methods

Method		D ₅₀ max/D ₅₀ min	D ₉₀ max/D ₉₀ min	D ₁₀ max/D ₁₀ min
Gravitational Sedimentation		2.3	5.2	3.5
Centrifugal Sedimentation		1.7	n.a.	n.a.
Light Scattering	(1)	2.1	4.9	1.3
	(2)	2.3	4.8	1.0

(1) Microtrac Instrument

(2) Cilas Instrument

In Table 4.1.5.3, the influence of the ultrasonic treatment on the D-values is demonstrated. As can be expected, lower values were obtained with a prolonged ultrasonic treatment.

Table 4.1.5.3 Influence of Ultrasonic Treatment of D-values in Microns

Lab #	IEA No.	Matl.	Method	Procedure ^a	D ₅₀	D ₉₀	D ₁₀
5	1584	SNR	(A)	A-11, 30 min, US	0.8	3.2	0.3
5	1584	SNR	(A)	A-11, 90 min, US	0.7	2.5	0.2
17	42	Si	(A)	A-11, 20 min, US	7.5	13.7	1.9
17	42	Si	(A)	A-11, 40 min, US	7.4	13.6	1.9
17	42	Si	(A)	A-11, 60 min, US	7.0	13.0	1.8
17	232	YSZ	(B)	H ₂ O, 3 min, US	2.2	8	0.4
17	232	YSZ	(B)	H ₂ O, 6 min, US	1.3	9	0.3
17	232	YSZ	(B)	H ₂ O, 12 min, US	1.4	9	0.3

Methods:

(A) Gravitational sedimentation

(B) Fraunhofer-Mie light scattering

US - Ultrasonication

^aA-11 is an mineral spirit based dispersion liquid in the series of SedisperseTM manufactured by Micromeritics, Inc.

In the case of zirconium oxide powder, constant values of d_{50} , d_{90} and d_{10} were obtained after a 6 minute ultrasonic treatment of the sample.

In Table 4.1.5.4, the influence of the dispersion medium on the D-values is shown. It appears that in this case the influence is even stronger than in the case of the dispersion time.

Table 4.1.5.4 Influence of Dispersion Medium on D-values in Microns

Lab	IEA No.	Powder	Method	Procedure	D ₅₀	D ₉₀	D ₁₀
1	449	SNR	(A)	A-11, 1 min, US	0.8	2.8	0.2
2	667	SNR	(A)	Deion. water, 0.4 g/100 mL A-11 pH 10; 0.2g/150mL; 3 min, US	1.2	5.7	0.4
17	1012	SiC		0.5% butylamine in H ₂ O; 3.5g/50mL; 10 min, US	0.6	1.0	0.2
22	7	SiC		A-11, 5 min, US	4.3	6.1	3.4

US - Ultrasonication

Conclusions

The data show a large scatter which is largely caused by the different methods which have been used for the preparation (dispersion) of the samples. In future interlaboratory studies, preparation methods should be standardized, and data should be obtained in such a way that a statistical treatment of various factors will be possible.

4.1.6 Permeametry - Fisher Sub-Sieve Size (R. Pompe)

Use and Significance

Permeametric determination of particle size is based on the measurement of pressure drop of a gas penetrating a bed of packed powder. It requires a uniformly packed, aggregate- and agglomerate-free bed to achieve homogeneous flow of gas. The calculated average particle diameter is dependent on the bed

porosity. The Fisher sub-sieve sizer is a convenient type of apparatus as it incorporates a device for compressing the bed to variable porosity levels. Fisher sub-sieve size (FSSS) measurement may provide an experimentally easily attained value of average particle diameter under the condition of a uniformly packed, homogeneous powder bed.

Instrument and Experimental Parameters

Permeametry measurements to determine FSSS were reported by two laboratories, No. 18 and No. 25. Lab 18 used Fisher Sub-sieve Sizer, Model 95, which was calibrated with Al_2O_3 (Alcoa A-12). Their results are summarized in Table 4.1.6.1 below.

The Fisher sub-sieve analyzer was calibrated with the synthetic ruby standard according to the Fisher procedure given in the instruction manual. In order to evaluate the test procedure as reported, an Al_2O_3 (Alcoa A-12) sample was examined, and steps were taken to determine the optimum porosity point. Measurements were performed at a porosity of 0.66, and the mean particle size was determined to be $3.1 \mu\text{m}$, see Table 4.1.6.2. This value compared favorably with that of $3.5 \mu\text{m}$ determined by using the Sedigraph.

Table 4.1.6.1 Summary of Fisher Sub-Sieve Size Results

Lab #	Material	No. of Values	Max./Min. Microns	Ave.
18	YSZ	4	8.0/0.70	-
25	SNR	1	-	0.6
25	SNT	2	1.11/0.95	1.03
25	SiC	1	-	0.46

Results and Discussion

The results obtained by Lab 18 and 25 are summarized in Table 4.1.6.1. The evaluation of particle size of the YSZ powder by Lab 18 is given in Table 4.1.6.2.

For the analysis of particle size in YSZ, a maximum porosity of 0.68 was obtained due to poor packing of fine powder. This density was not sufficient to yield a reasonable particle size value (expected to be $0.7 \mu\text{m}$). It was concluded that this test was not appropriate for this powder.

Conclusions

The use of permeametry for submicrometer sized powders, such as the YSZ powder, was considered not to be appropriate.

Table 4.1.6.2

Permeametry Data for YSZ Powder

A-12 Al ₂ O ₃ Reference		YSZ ZrO ₂	
Porosity	Mean Part. Size (μm)	Porosity	Mean Size (μm)
0.80	2.29	0.80	8.0
0.75	2.82	0.75	4.5
0.70	3.42	0.70	1.15
0.65	3.95	0.68	0.70
0.60	3.01		
0.55	2.93		
0.68	3.23		
0.66	3.48		
0.64	3.38		
0.62	3.09		
0.66	3.12		

4.2. STATISTICAL DATA

4.2.1. Mathematical Functions

Results are given for the experimental mean, usually the D_{50} ; 90% finer size, D_{90} ; 10% finer size, D_{10} ; and spread ratio, calculated as $(D_{90} - D_{10})/D_{50}$.

Under the assumption of a lognormal distribution, the geometric standard deviation (σ_g) is given by

$$\sigma_g = \sqrt{(D_{84}/D_{16})} \quad , \quad (4.2.1)$$

where D_{84} is the size at which 84% of the particles are finer and D_{16} is the size at which 16% of the particles are finer. The geometric standard deviation can be estimated from the spread ratio. Using the assumption of lognormality, relationship between σ_g and the spread ratio is as follows:

$$\sigma_g = \exp\{0.77185 \cdot \sinh^{-1}([D_{90} - D_{10}]/2D_{50})\} \quad (4.2.2)$$

4.2.2. Gravitational Sedimentation (Sedigraph)

4.2.2.1. Silicon Nitride Reference (SNR) Powder

Table 4.2.2.1.1 Experimental Mean, D_{50} , μm

Material: Silicon Nitride, Reference Powder (Figure 26)
 Property: EXPTLMEAN
 Method: 9

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	11	0.87	1.12	0.98	0.08
3	4	1.25	1.40	1.32	0.06
4	14	0.42	0.86	0.68	0.13
6	2	0.80	0.95	0.87	0.10
9	8	1.10	1.20	1.14	0.03
15	1	1.00	1.00	1.00	0.00
16	2	0.77	0.78	0.77	0.00
17	6	0.68	0.90	0.76	0.08
18	1	0.80	0.80	0.80	0.00
23	39	0.61	1.07	0.78	0.10
Means	10	0.68	1.32	0.91	0.20

Table 4.2.2.1.2 Size at 90% Finer, D_{90} , μm

Material: Silicon Nitride, Reference Powder
 Property: D_{90}
 Method: 9

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	11	4.10	7.70	5.87	1.23
3	4	3.20	7.30	4.62	1.82
4	14	1.70	4.10	2.47	0.65
6	2	2.80	5.00	3.90	1.55
9	8	4.85	6.20	5.53	0.51
15	1	5.00	5.00	5.00	0.00
17	5	3.05	3.50	3.28	0.19
18	1	4.90	4.90	4.90	0.00
23	39	1.70	6.40	3.51	0.76
Means	9	2.47	5.87	4.34	1.12

Table 4.2.2.1.3 Size at 10% Finer, D_{10} , μm

Material: Silicon Nitride, Reference Powder
 Property: D_{10}
 Method: 9

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	11	0.24	0.34	0.27	0.03
3	4	0.61	0.67	0.63	0.02
4	9	0.24	0.37	0.32	0.05
6	2	0.17	0.35	0.26	0.12
9	8	0.34	0.37	0.34	0.01
15	1	0.30	0.30	0.30	0.00
17	5	0.20	0.33	0.25	0.05
18	1	0.23	0.23	0.23	0.00
23	24	0.18	0.36	0.25	0.04
Means	9	0.23	0.63	0.32	0.12

Table 4.2.2.1.4 Spread Ratio of Size Distribution, $(D_{90} - D_{10})/D_{50}$

Material: Silicon Nitride, Reference Powder
 Property: $(D_{90} - D_{10})/D_{50}$
 Method: 9

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	11	4.38	7.77	5.66	0.94
3	4	2.07	4.95	2.99	1.32
4	9	2.25	4.35	3.10	0.63
6	2	3.29	4.89	4.09	1.13
9	8	4.07	4.86	4.51	0.32
15	1	4.70	4.70	4.70	0.00
17	5	3.56	4.32	3.91	0.33
18	1	5.84	5.84	5.84	0.00
23	24	2.37	6.57	3.80	0.91
Means	9	2.99	5.84	4.29	1.00

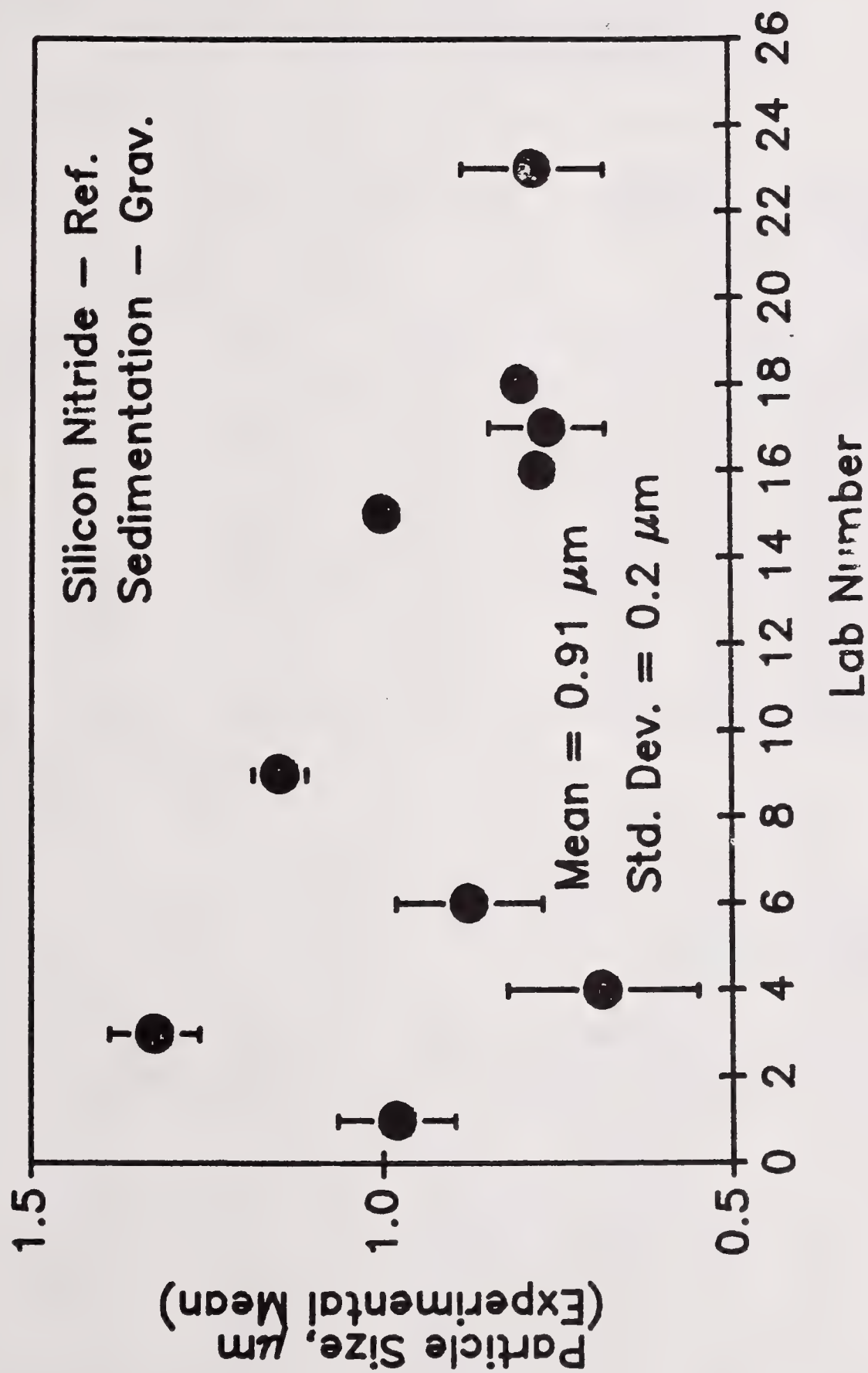


Figure 26. Experimental mean particle size of SNR powder by gravitational sedimentation.

4.2.2. Gravitational Sedimentation (Sedigraph)

4.2.2.2 Silicon Nitride Test (SNT) Powder

Table 4.2.2.2.1 Experimental Mean, D_{50} , μm

Material: Silicon Nitride, Test Powder (Figure 27)
 Property: EXPTLMEAN
 Method: 9

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	2	0.55	0.58	0.56	0.02
3	2	0.69	0.69	0.69	0.00
4	5	0.48	0.53	0.49	0.02
6	1	0.64	0.64	0.64	0.00
9	10	0.62	0.72	0.69	0.03
15	1	0.42	0.42	0.42	0.00
16	2	0.42	0.54	0.48	0.08
17	6	0.54	0.67	0.60	0.04
18	1	0.27	0.27	0.27	0.00
23	11	0.35	0.50	0.42	0.03
Means	10	0.27	0.69	0.52	0.13

Table 4.2.2.2.2 Size at 90% Finer, D_{90} , μm

Material: Silicon Nitride, Test Powder
 Property: D_{90}
 Method: 9

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	2	0.91	0.98	0.94	0.04
3	2	1.10	1.15	1.12	0.03
4	5	0.70	0.84	0.74	0.05
6	1	1.03	1.03	1.03	0.00
9	10	1.05	1.12	1.09	0.02
15	1	0.90	0.90	0.90	0.00
17	6	0.82	1.04	0.93	0.08
18	1	0.64	0.64	0.64	0.00
23	11	0.54	1.00	0.77	0.13
Means	9	0.64	1.12	0.90	0.16

Table 4.2.2.2.3 Size at 10% Finer, D_{10} , μm

Material: Silicon Nitride, Test Powder
 Property: D_{10}
 Method: 9

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	2	0.33	0.36	0.34	0.02
3	2	0.41	0.43	0.42	0.01
4	5	0.27	0.32	0.29	0.02
6	1	0.38	0.38	0.38	0.00
9	10	0.32	0.44	0.37	0.03
15	1	0.33	0.33	0.33	0.00
17	6	0.36	0.42	0.38	0.02
23	11	0.19	0.28	0.23	0.02
Means	8	0.23	0.42	0.34	0.05

Table 4.2.2.2.4 Spread Ratio of Size Distribution, $(D_{90} - D_{10})/D_{50}$

Material: Silicon Nitride, Test Powder
 Property: $(D_{90} - D_{10})/D_{50}$
 Method: 9

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	2	1.05	1.08	1.06	0.02
3	2	1.00	1.04	1.02	0.02
4	5	0.83	1.02	0.90	0.06
6	1	1.02	1.02	1.02	0.00
9	10	0.92	1.18	1.04	0.08
15	1	1.36	1.36	1.36	0.00
17	6	0.85	0.99	0.90	0.06
23	11	1.00	1.72	1.26	0.21
Means	8	0.90	1.36	1.07	0.16

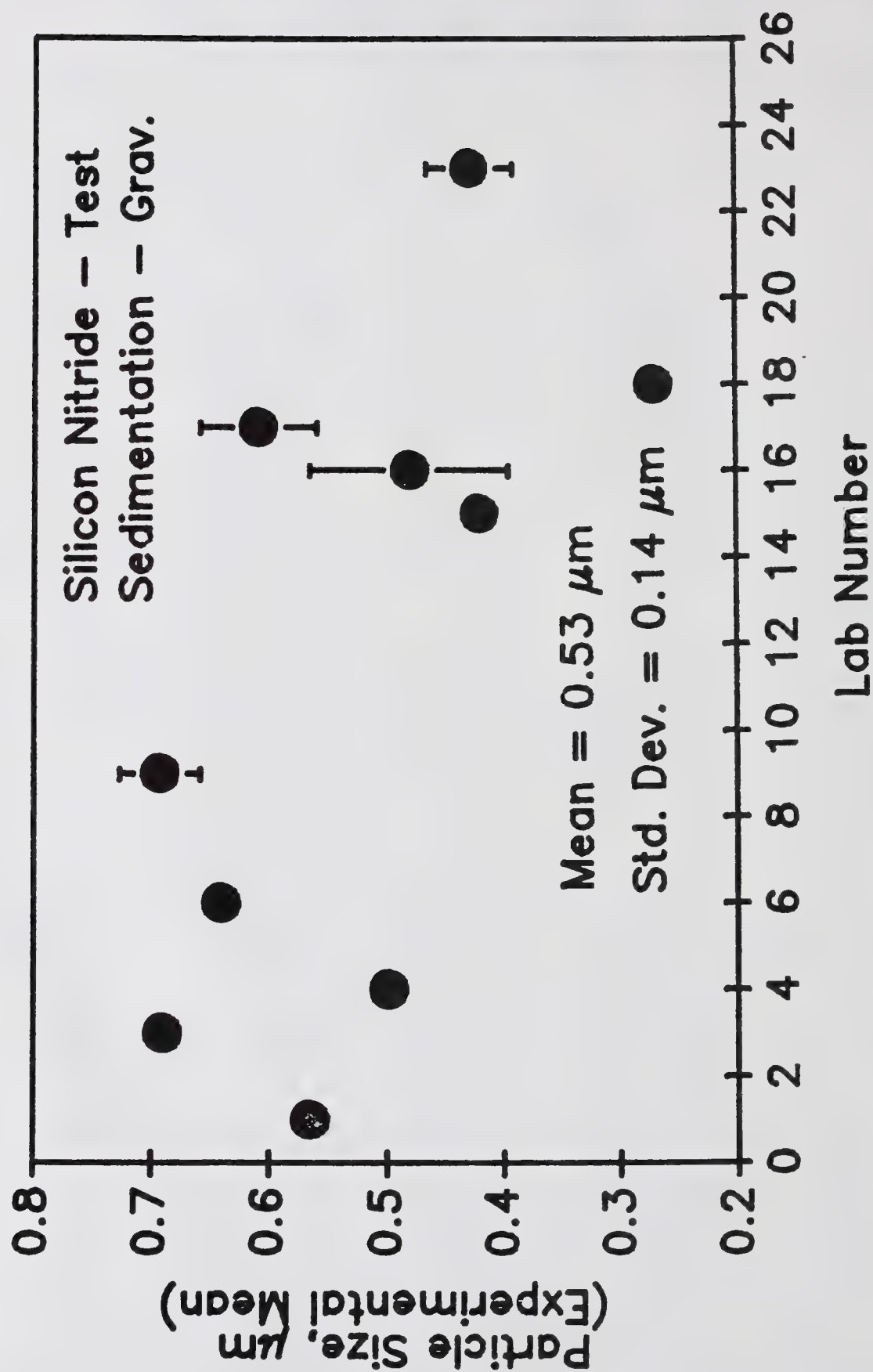


Figure 27. Experimental mean particle size of SNT powder by gravitational sedimentation.

4.2.2. Gravitational Sedimentation (Sedigraph)

4.2.2.3 Silicon Carbide (SiC) Powder

Table 4.2.2.3.1 Experimental Mean, D_{50} , μm

Material: Silicon Carbide (Figure 28)
 Property: EXPTLMEAN
 Method: 9

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	3	0.75	0.76	0.75	0.00
3	13	4.00	5.00	4.41	0.31
4	3	0.49	0.65	0.57	0.08
6	1	0.82	0.82	0.82	0.00
9	7	0.52	0.59	0.56	0.02
10	1	0.90	0.90	0.90	0.00
15	1	0.55	0.55	0.55	0.00
16	2	0.46	0.53	0.49	0.04
23	21	0.55	1.03	0.74	0.13
Means	9	0.49	4.41	1.09	1.25

Table 4.2.2.3.2 Size at 90% Finer, D_{90} , μm

Material: Silicon Carbide
 Property: D_{90}
 Method: 9

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	3	1.20	1.50	1.30	0.17
3	13	5.80	7.20	6.33	0.39
4	3	0.80	0.95	0.87	0.07
6	1	1.30	1.30	1.30	0.00
9	7	1.05	1.25	1.12	0.06
10	1	1.50	1.50	1.50	0.00
15	1	1.00	1.00	1.00	0.00
16	2	0.85	1.00	0.92	0.10
23	21	0.98	2.15	1.45	0.29
Means	9	0.87	6.33	1.75	1.72

Table 4.2.2.3.3 Size at 10% Finer, D_{10} , μm

Material: Silicon Carbide
 Property: D_{10}
 Method: 9

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	3	0.52	0.53	0.52	0.00
3	13	2.80	4.40	3.70	0.44
4	3	0.36	0.46	0.41	0.05
6	1	0.62	0.62	0.62	0.00
9	7	0.19	0.21	0.20	0.00
15	1	0.20	0.20	0.20	0.00
16	2	0.19	0.20	0.19	0.00
23	21	0.35	0.55	0.47	0.06
Means	8	0.19	3.70	0.79	1.18

Table 4.2.2.3.4 Spread Ratio of Size Distribution, $(D_{90} - D_{10})/D_{50}$

Material: Silicon Carbide
 Property: $(D_{90} - D_{10})/D_{50}$
 Method: 9

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	3	0.89	1.28	1.02	0.22
3	13	0.46	0.78	0.60	0.10
4	3	0.75	0.90	0.81	0.07
6	1	0.83	0.83	0.83	0.00
9	7	1.54	1.80	1.62	0.08
15	1	1.45	1.45	1.45	0.00
16	2	1.41	1.53	1.47	0.08
23	21	0.99	1.69	1.32	0.20
Means	8	0.60	1.62	1.14	0.37

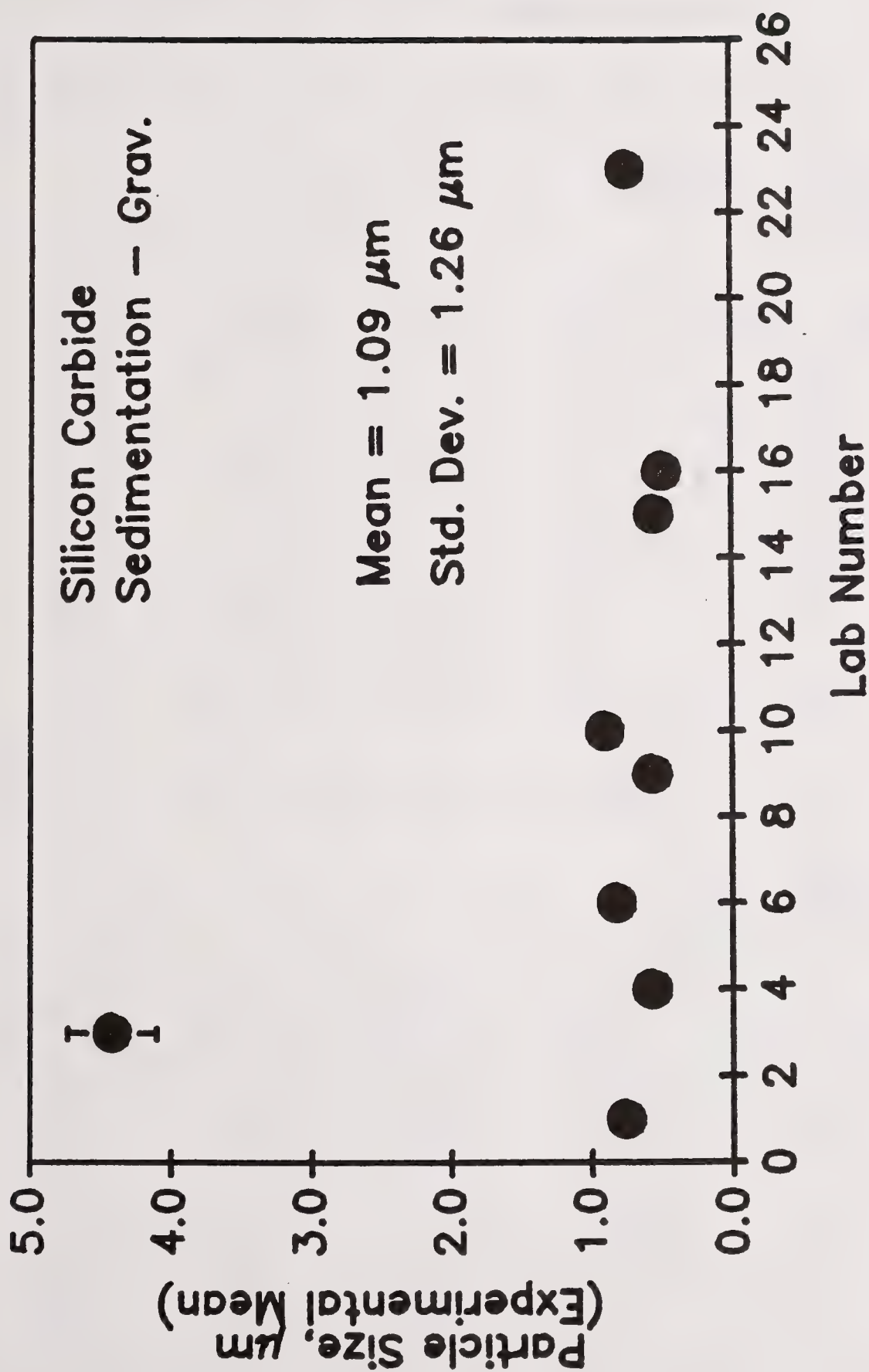


Figure 28. Experimental mean particle size of SiC powder by gravitational sedimentation.

4.2.2. Gravitational Sedimentation (Sedigraph)

4.2.2.4 Silicon (Si) Powder

Table 4.2.2.4.1 Experimental Mean, D_{50} , μm

Material: Silicon (Figure 29)
 Property: EXPTLMEAN
 Method: 9

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	3	6.70	6.80	6.73	0.05
3	11	5.40	6.30	5.81	0.21
4	6	5.40	5.90	5.63	0.23
6	1	5.80	5.80	5.80	0.00
9	6	6.50	6.80	6.61	0.11
15	1	6.20	6.20	6.20	0.00
16	2	5.58	5.65	5.61	0.04
17	13	6.70	7.60	7.25	0.30
18	1	5.60	5.60	5.60	0.00
23	11	5.70	6.90	6.32	0.33
Means	10	5.60	7.25	6.15	0.56

Table 4.2.2.4.2 Size at 90% Finer, D_{90} , μm

Material: Silicon
 Property: D_{90}
 Method: 9

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	3	12.00	12.80	12.43	0.40
3	11	9.50	10.80	10.45	0.34
4	6	10.10	10.80	10.46	0.28
6	1	10.07	10.07	10.07	0.00
9	6	11.50	12.30	11.71	0.34
15	1	10.50	10.50	10.50	0.00
16	2	9.52	9.67	9.59	0.10
17	13	12.00	14.20	13.26	0.59
18	1	10.00	10.00	10.00	0.00
23	11	12.00	14.20	12.94	0.67
Means	10	9.59	13.26	11.14	1.33

Table 4.2.2.4.3 Size at 10% Finer, D_{10} , μm

Material: Silicon
 Property: D_{10}
 Method: 9

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	3	1.75	1.90	1.83	0.07
3	11	1.40	2.30	1.85	0.26
4	6	1.01	1.55	1.31	0.23
6	1	1.55	1.55	1.55	0.00
9	6	2.00	2.50	2.27	0.22
15	1	2.00	2.00	2.00	0.00
16	2	2.03	2.13	2.08	0.07
17	12	1.55	2.00	1.84	0.16
18	1	1.50	1.50	1.50	0.00
23	11	1.30	2.30	1.73	0.34
Means	10	1.31	2.27	1.79	0.28

Table 4.2.2.4.4 Spread Ratio of Size Distribution, $(D_{90} - D_{10})/D_{50}$

Material: Silicon
 Property: $(D_{90} - D_{10})/D_{50}$
 Method: 9

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	3	1.51	1.61	1.57	0.05
3	11	1.30	1.57	1.47	0.07
4	6	1.57	1.72	1.62	0.06
6	1	1.47	1.47	1.47	0.00
9	6	1.37	1.49	1.42	0.04
15	1	1.37	1.37	1.37	0.00
16	2	1.32	1.35	1.33	0.02
17	12	1.52	1.63	1.57	0.03
18	1	1.52	1.52	1.52	0.00
23	11	1.46	2.25	1.78	0.20
Means	10	1.33	1.78	1.51	0.13

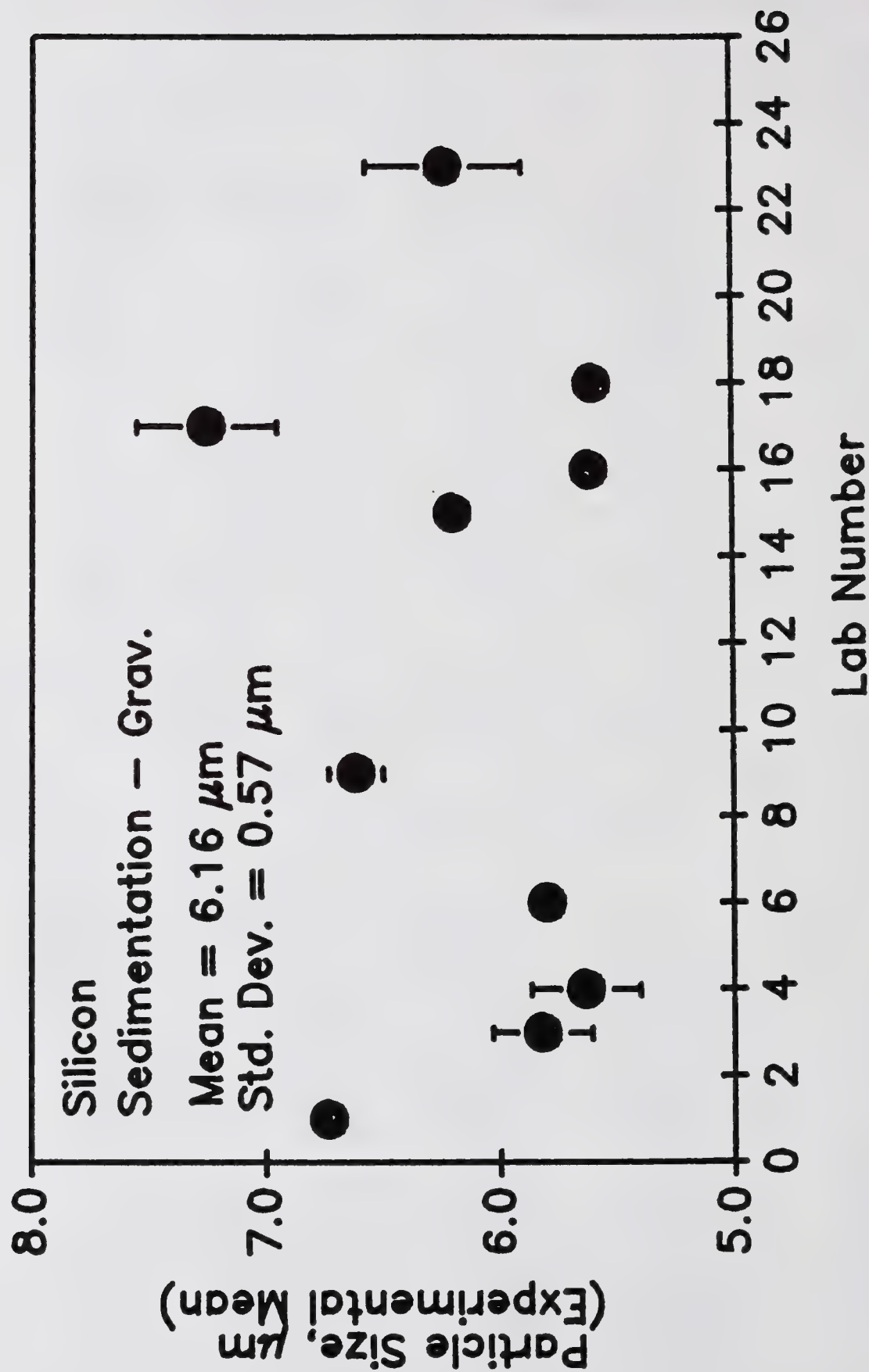


Figure 29. Experimental mean particle size of Si powder by gravitational sedimentation.

4.2.2. Gravitational Sedimentation (Sedigraph)

4.2.2.5 Yttria-Zirconia (YSZ) Powder

Table 4.2.2.5.1 Experimental Mean, D_{50} , μm

Material: Zirconia (Figure 30)
 Property: EXPTLMEAN
 Method: 9

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
4	6	0.15	0.32	0.20	0.07
6	1	0.20	0.20	0.20	0.00
15	1	0.20	0.20	0.20	0.00
17	21	0.14	0.76	0.25	0.15
18	5	0.15	0.50	0.22	0.15
23	2	0.15	0.18	0.16	0.02
Means	6	0.16	0.25	0.20	0.02

Table 4.2.2.5.2 Size at 90% Finer, D_{90} , μm

Material: Zirconia
 Property: D_{90}
 Method: 9

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
4	8	2.20	4.00	3.02	0.55
6	1	3.20	3.20	3.20	0.00
15	1	4.00	4.00	4.00	0.00
17	6	3.70	8.50	6.11	1.88
18	5	10.50	14.00	11.70	1.39
Means	5	3.02	11.70	5.60	3.62

Table 4.2.2.5.3 Size at 10% Finer, D_{10} , μm

Material: Zirconia
 Property: D_{10}
 Method: 9

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
17	1	0.16	0.16	0.16	0.00
Means	1	0.16	0.16	0.16	0.00

Table 4.2.2.5.4 Spread Ratio of Size Distribution, $(D_{90} - D_{10})/D_{50}$

Material: Zirconia
 Property: $(D_{90} - D_{10})/D_{50}$
 Method: 9

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
17	1	20.78	20.78	20.78	0.00
Means	1	20.78	20.78	20.78	0.00

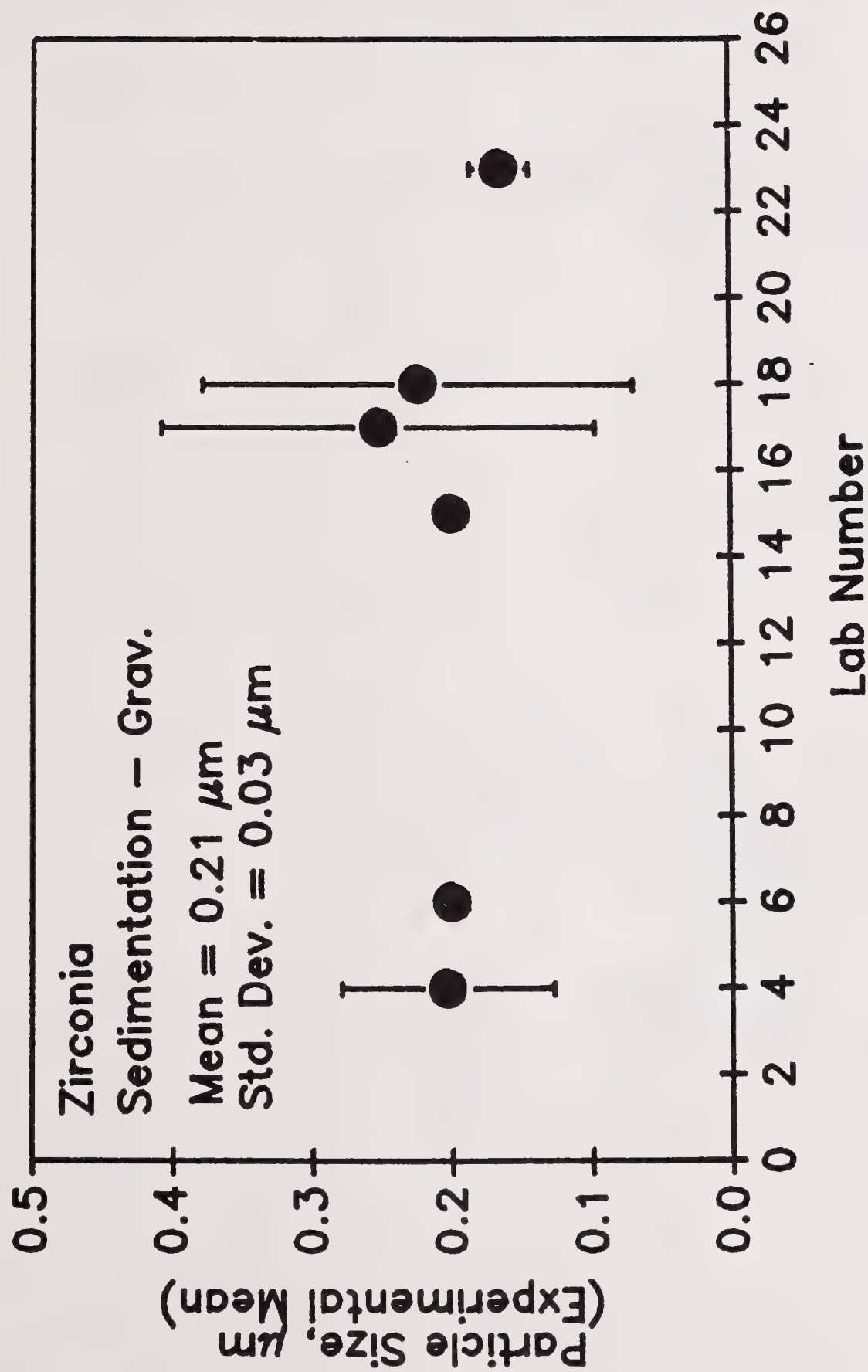


Figure 30. Experimental mean particle size of YSZ powder by gravitational sedimentation.

4.2.3. Centrifugal Sedimentation

4.2.3.1 Silicon Nitride Reference Powder

Table 4.2.3.1.1 Experimental Mean, D_{50} , μm

Material: Silicon Nitride, Reference Powder (Figure 31)
 Property: EXPTLMEAN
 Method: 10

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	9	0.91	1.14	1.02	0.06
5	3	0.67	0.70	0.68	0.01
11	1	0.57	0.57	0.57	0.00
13	20	1.21	1.67	1.40	0.11
14	1	0.98	0.98	0.98	0.00
19	1	1.51	1.51	1.51	0.00
Means	6	0.57	1.51	1.02	0.37

Table 4.2.3.2.2 Size at 90% Finer, D_{90} , μm

Material: Silicon Nitride, Reference Powder
 Property: D_{90}
 Method: 10

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
5	3	3.80	5.00	4.26	0.64
11	1	2.90	2.90	2.90	0.00
14	1	4.30	4.30	4.30	0.00
Means	3	2.90	4.30	3.82	0.79

Table 4.2.3.1.3 Size at 10% Finer, D_{10} , μm

Material: Silicon Nitride, Reference Powder
 Property: D_{10}
 Method: 10

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
5	3	0.26	0.30	0.28	0.02
11	1	0.20	0.20	0.20	0.00
14	1	0.29	0.29	0.29	0.00
Means	3	0.20	0.29	0.25	0.05

Table 4.2.3.1.4 Spread Ratio of Size Distribution, $(D_{90} - D_{10})/D_{50}$

Material: Silicon Nitride, Reference Powder
 Property: $(D_{90} - D_{10})/D_{50}$
 Method: 10

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
5	3	5.22	6.77	5.81	0.83
11	1	4.74	4.74	4.74	0.00
14	1	4.09	4.09	4.09	0.00
Means	3	4.09	5.81	4.88	0.87

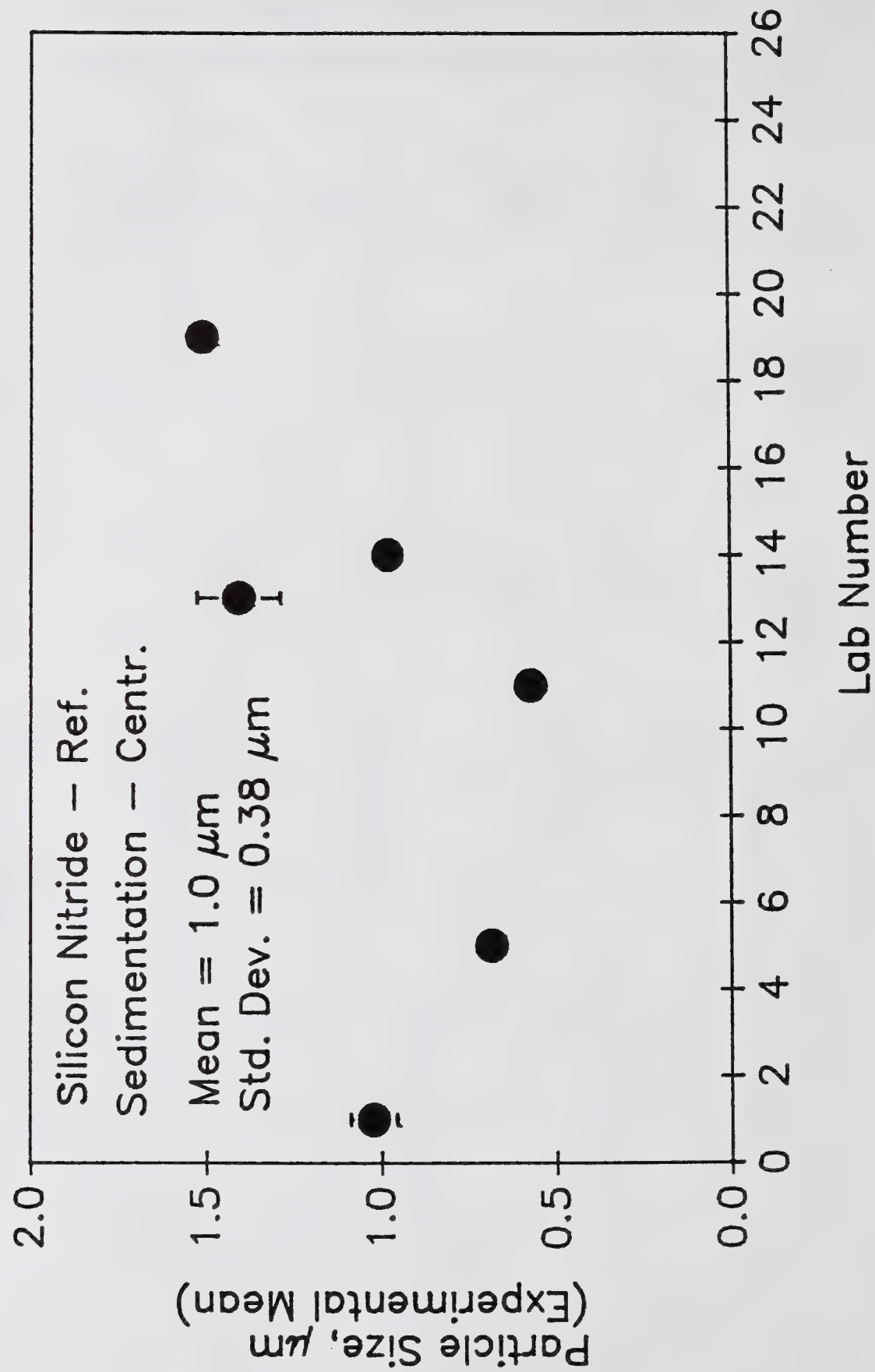


Figure 31. Experimental mean particle size of SNR powder by centrifugal sedimentation.

4.2.3. Centrifugal Sedimentation

4.2.3.2 Silicon Nitride Test Powder

Table 4.2.3.2.1 Experimental Mean

Material: Silicon Nitride, Test Powder (Figure 32)
 Property: EXPTLMEAN
 Method: 10

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	2	0.77	0.78	0.77	0.00
5	4	0.53	0.56	0.53	0.01
14	3	0.55	0.66	0.59	0.06
19	10	0.77	1.10	0.88	0.09
Means	4	0.53	0.88	0.69	0.16

Table 4.2.3.2.2 Size at 90% Finer, D_{90} , μm

Material: Silicon Nitride, Test Powder
 Property: D_{90}
 Method: 10

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
5	4	1.14	1.89	1.35	0.35
14	3	1.20	1.81	1.41	0.34
Means	2	1.35	1.41	1.38	0.04

Table 4.2.3.2.3 Size at 10% Finer, D_{10} , μm

Material: Silicon Nitride, Test Powder
 Property: D_{10}
 Method: 10

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
5	4	0.24	0.25	0.24	0.00
14	3	0.27	0.34	0.29	0.04
Means	2	0.24	0.29	0.27	0.03

Table 4.2.3.2.4 Spread Ratio of Size Distribution, $(D_{90} - D_{10})/D_{50}$

Material: Silicon Nitride, Test Powder
 Property: $(D_{90} - D_{10})/D_{50}$
 Method: 10

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
5	4	1.68	2.95	2.04	0.60
14	3	1.66	2.23	1.87	0.31
Means	2	1.87	2.04	1.95	0.11

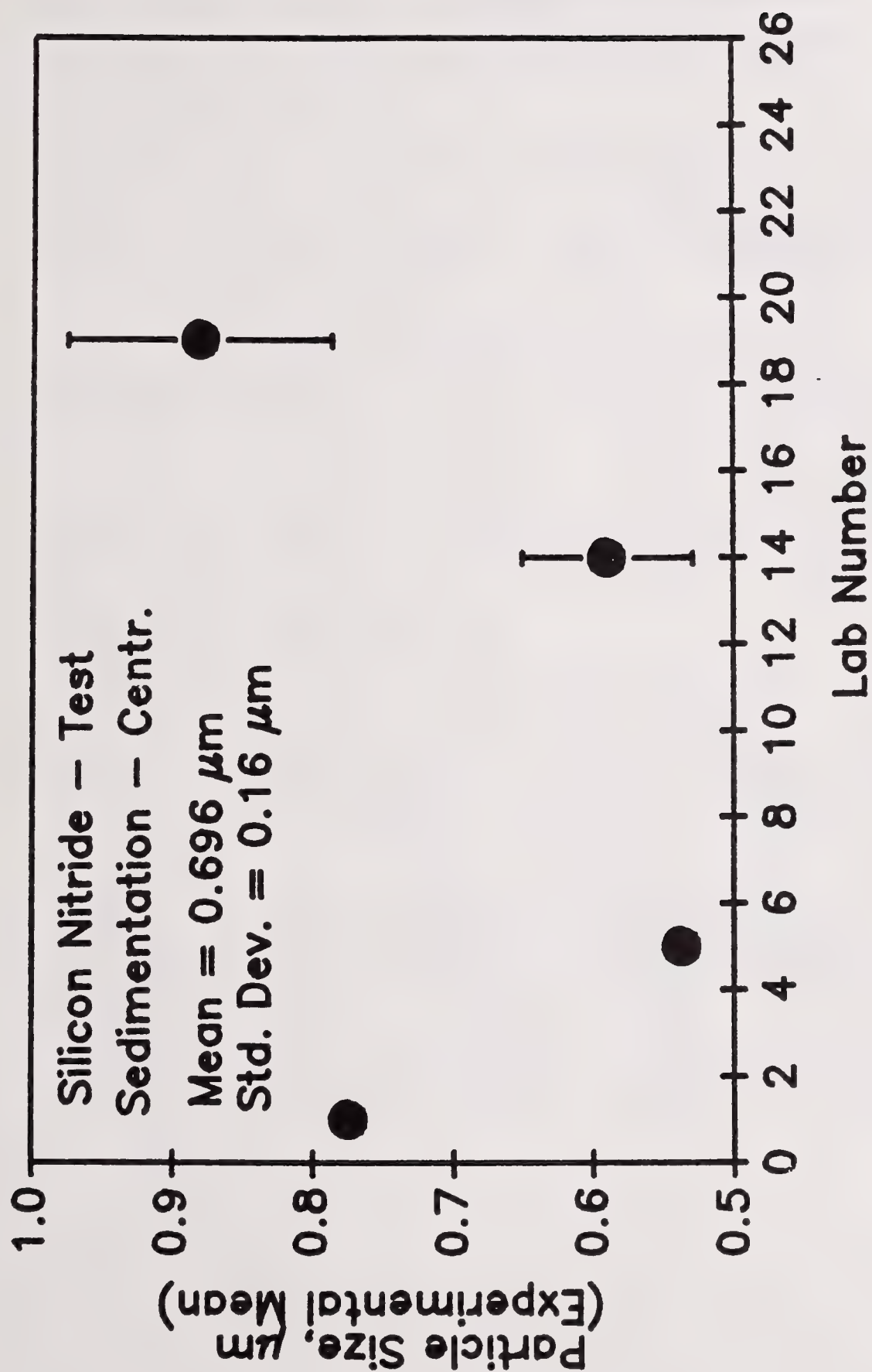


Figure 32. Experimental mean particle size of SNT powder by centrifugal sedimentation.

4.2.3. Centrifugal Sedimentation

4.2.3.3 Silicon Carbide Powder

Table 4.2.3.3.1 Experimental Mean, D_{50} , μm

Material: Silicon Carbide
 Property: EXPTLMEAN
 Method: 10

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	6	0.58	0.83	0.69	0.08
2	1	0.40	0.40	0.40	0.00
13	10	0.63	0.71	0.66	0.02
Means	3	0.40	0.69	0.58	0.16

Table 4.2.3.3.2 Size at 90% Finer, D_{90} , μm

Material: Silicon Carbide
 Property: D_{90}
 Method: 10

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	6	1.50	2.06	1.78	0.18
2	1	0.86	0.86	0.86	0.00
Means	2	0.86	1.78	1.32	0.65

Table 4.2.3.3.3 Size at 10% Finer, D_{10} , μm

Material: Silicon Carbide
 Property: D_{10}
 Method: 10

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	6	0.29	0.45	0.38	0.06
2	1	0.11	0.11	0.11	0.00
Means	2	0.11	0.38	0.24	0.19

Table 4.2.3.3.4 Spread Ratio of Size Distribution, $(D_{90} - D_{10})/D_{50}$

Material: Silicon Carbide
 Property: $(D_{90} - D_{10})/D_{50}$
 Method: 10

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	6	1.48	2.47	2.05	0.34
2	1	1.87	1.87	1.87	0.00
Means	2	1.87	2.05	1.96	0.13

4.2.3. Centrifugal Sedimentation

4.2.3.4 Silicon Powder

Table 4.2.3.4.1 Experimental Mean, D_{50} , μm

Material: Silicon (Figure 33)
 Property: EXPTLMEAN
 Method: 10

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	3	4.09	4.29	4.17	0.10
2	1	6.60	6.60	6.60	0.00
5	2	4.90	5.00	4.95	0.07
19	9	4.62	6.05	5.08	0.44
Means	4	4.17	6.60	5.20	1.01

Table 4.2.3.4.2 Size at 90% Finer, D_{90} , μm

Material: Silicon
 Property: D_{90}
 Method: 10

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	3	7.06	7.49	7.30	0.21
2	1	9.80	9.80	9.80	0.00
5	2	11.70	11.80	11.75	0.07
Means	3	7.30	11.75	9.61	2.23

Table 4.2.3.4.3 Size at 10% Finer, D_{10} , μm

Material: Silicon
 Property: D_{10}
 Method: 10

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	3	1.40	1.95	1.76	0.31
2	1	0.43	0.43	0.43	0.00
5	2	0.56	0.56	0.56	0.00
Means	3	0.43	1.76	0.91	0.73

Table 4.2.3.4.4 Spread Ratio of Size Distribution, $(D_{90} - D_{10})/D_{50}$

Material: Silicon
 Property: $(D_{90} - D_{10})/D_{50}$
 Method: 10

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	3	1.19	1.47	1.32	0.14
2	1	1.42	1.42	1.42	0.00
5	2	2.25	2.27	2.26	0.01
Means	3	1.32	2.26	1.66	0.51

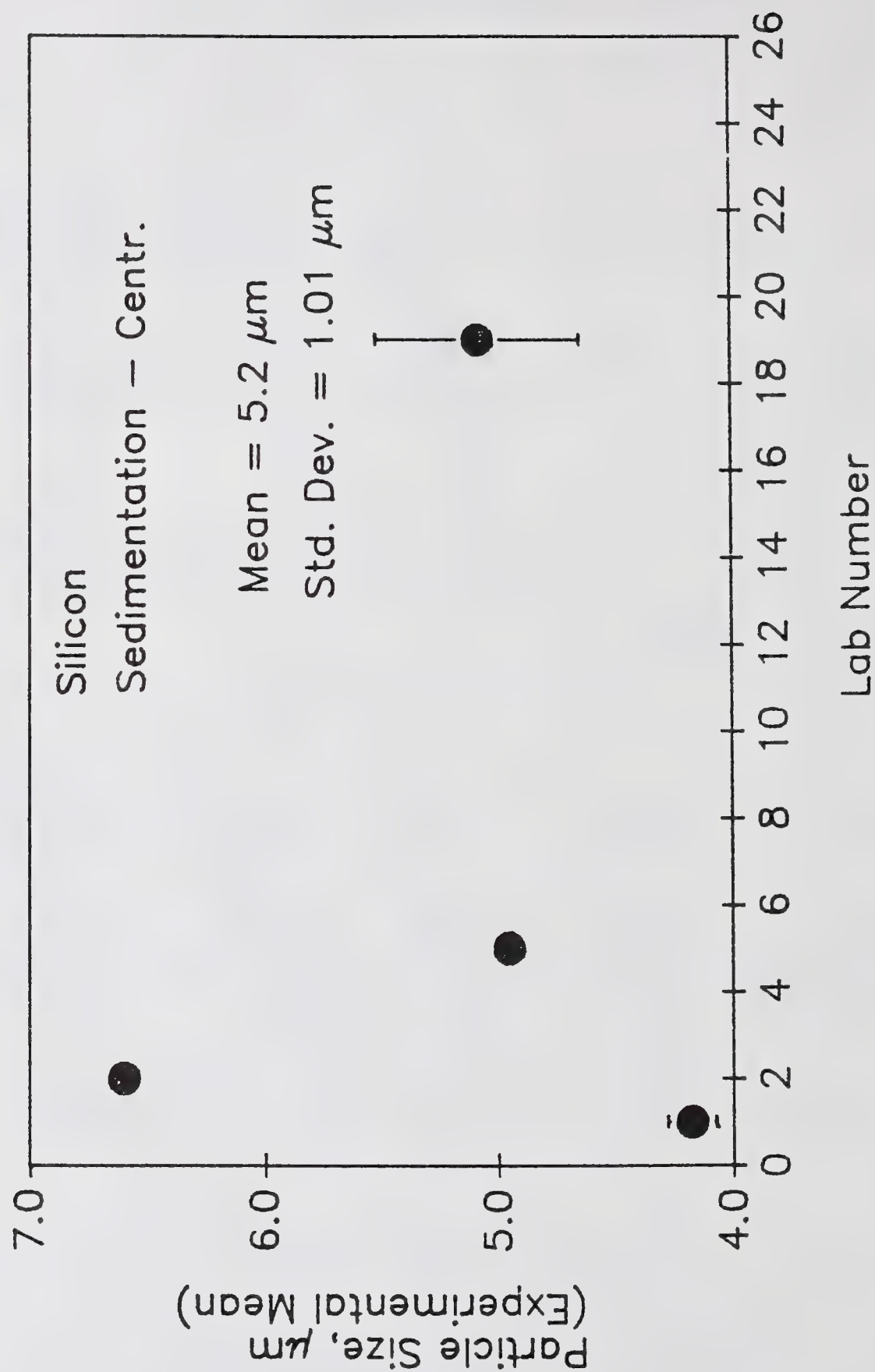


Figure 33. Experimental mean particle size of Si powder by centrifugal sedimentation.

4.2.3. Centrifugal Sedimentation

4.2.3.5 Yttria-Zirconia Powder

Table 4.2.3.5.1 Experimental Mean, D_{50} , μm

Material: Zirconia (Figure 34)
 Property: EXPTLMEAN
 Method: 10

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	6	1.38	1.62	1.49	0.08
11	1	0.56	0.56	0.56	0.00
14	1	1.73	1.73	1.73	0.00
19	18	1.21	1.69	1.47	0.11
Means	4	0.56	1.73	1.31	0.51

Table 4.2.3.5.2 Size at 90% Finer, D_{90} , μm

Material: Zirconia
 Property: D_{90}
 Method: 10

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
11	1	2.90	2.90	2.90	0.00
14	1	7.30	7.30	7.30	0.00
Means	2	2.90	7.30	5.10	3.11

Table 4.2.3.5.3 Size at 10% Finer, D_{10} , μm

Material: Zirconia
 Property: D_{10}
 Method: 10

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	1	0.18	0.18	0.18	0.00
14	1	0.29	0.29	0.29	0.00
Means	2	0.18	0.29	0.23	0.07

Table 4.2.3.5.4 Spread Ratio of Size Distribution, $(D_{90} - D_{10})/D_{50}$

Material: Zirconia
 Property: $(D_{90} - D_{10})/D_{50}$
 Method: 10

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
11	1	4.86	4.86	4.86	0.00
14	1	4.05	4.05	4.05	0.00
Means	2	4.05	4.86	4.45	0.57

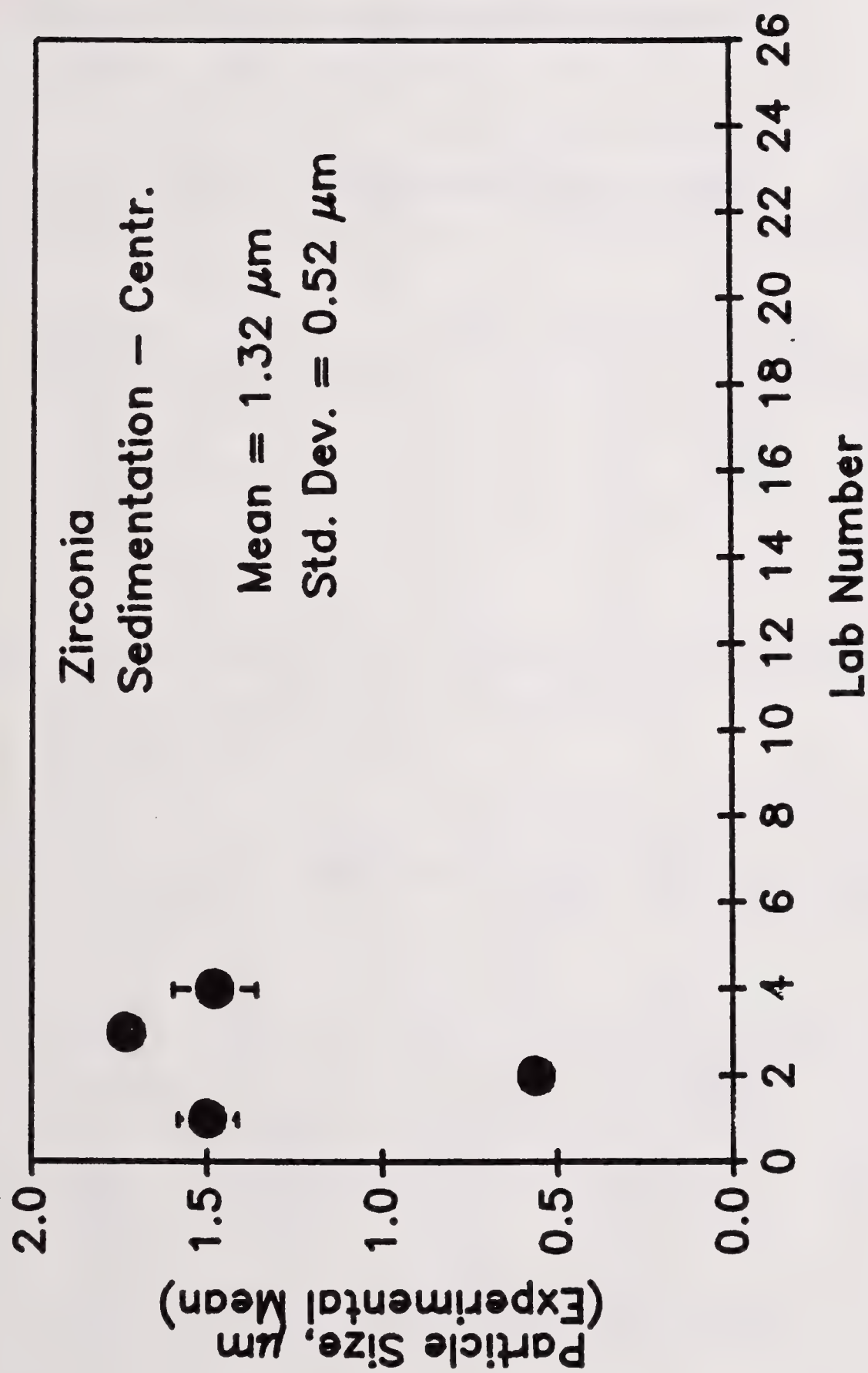


Figure 34. Experimental mean particle size of YSZ powder by centrifugal sedimentation.

4.2.4. Light-Scattering Fraunhofer-Mie Type Instruments

4.2.4.1 Silicon Nitride Reference Powder

Table 4.2.4.1.1 Experimental Mean, D_{50} , μm

Material: Silicon Nitride, Reference Powder (Figure 35)
 Property: ExPTLMEAN
 Method: 13

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
7	5	1.33	1.35	1.34	0.00
8	28	0.98	1.25	1.12	0.06
9	24	1.14	1.35	1.23	0.05
14	2	0.75	0.77	0.76	0.01
20	7	1.09	1.52	1.34	0.13
21	1	0.79	0.79	0.79	0.00
22	36	0.72	0.90	0.80	0.06
24	2	0.40	0.85	0.62	0.31
25	3	0.73	0.80	0.76	0.03
Means	9	0.62	1.34	0.97	0.28

Table 4.2.4.1.2 Size at 90% Finer, D_{90} , μm

Material: Silicon Nitride, Reference Powder
 Property: D_{90}
 Method: 13

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
8	28	3.45	7.33	5.19	0.86
9	24	5.44	7.35	6.46	0.63
14	1	2.00	2.00	2.00	0.00
20	7	5.15	11.52	8.62	2.10
21	1	3.20	3.20	3.20	0.00
22	36	1.86	2.58	2.26	0.22
24	2	2.90	9.60	6.25	4.73
25	3	2.61	2.91	2.76	0.15
Means	8	2.00	8.62	4.59	2.39

Table 4.2.4.1.3 Size at 10% Finer, D_{10} , μm

Material: Silicon Nitride, Reference Powder
 Property: D_{10}
 Method: 13

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
8	28	0.40	0.43	0.40	0.00
9	24	0.34	0.37	0.35	0.00
14	1	0.31	0.31	0.31	0.00
20	7	0.37	0.40	0.38	0.00
21	1	0.28	0.28	0.28	0.00
22	36	0.31	0.36	0.33	0.01
24	2	0.10	0.24	0.17	0.09
25	3	0.32	0.33	0.32	0.00
Means	8	0.17	0.40	0.32	0.07

Table 4.2.4.1.4 Spread Ratio of Size Distribution, $(D_{90} - D_{10})/D_{50}$

Material: Silicon Nitride, Reference Powder
 Property: $(D_{90} - D_{10})/D_{50}$
 Method: 13

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
8	28	3.10	5.57	4.22	0.55
9	24	4.36	5.44	4.91	0.34
14	1	2.25	2.25	2.25	0.00
20	7	4.39	7.67	6.03	1.03
21	1	3.70	3.70	3.70	0.00
22	36	2.12	2.71	2.40	0.18
24	2	7.00	11.01	9.00	2.83
25	3	3.14	3.23	3.18	0.04
Means	8	2.25	9.00	4.46	2.22

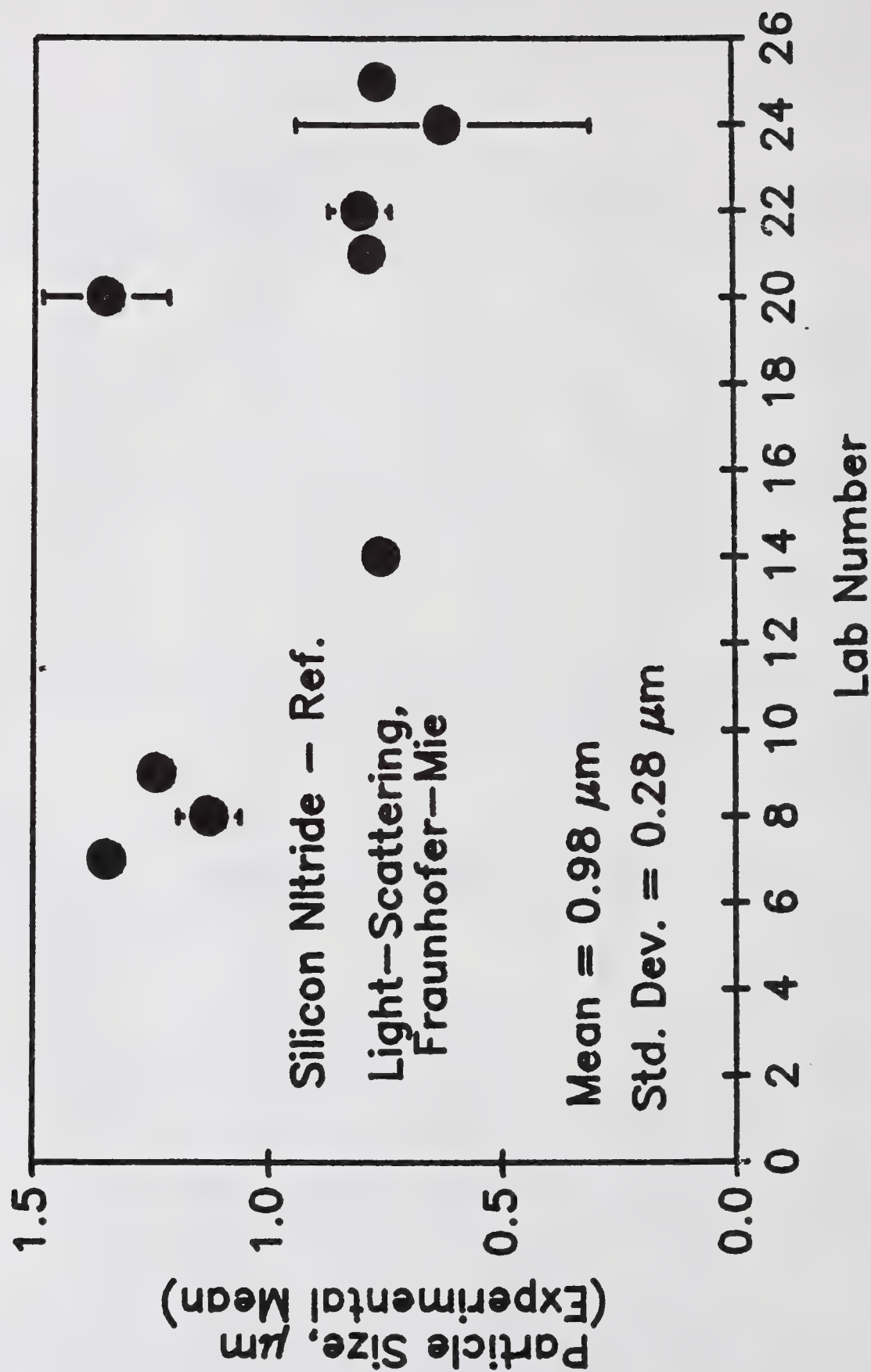


Figure 35. Experimental mean particle size of SNR powder by light-scattering, Fraunhofer-Mie type instrument.

4.2.4. Light Scattering Fraunhofer-Mie Type Instruments

4.2.4.2 Silicon Nitride Test Powder

Table 4.2.4.2.1 Experimental Mean, D_{50} , μm

Material: Silicon Nitride, Test Powder (Figure 36)
 Property: EXPTLMEAN
 Method: 13

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
7	10	0.61	0.63	0.62	0.00
8	35	0.65	0.72	0.67	0.02
9	54	0.60	0.63	0.61	0.00
14	4	0.52	0.57	0.54	0.02
20	3	0.58	0.58	0.58	0.00
22	30	0.56	0.60	0.57	0.01
24	4	0.69	0.77	0.73	0.03
25	3	0.54	0.55	0.54	0.00
Means	8	0.54	0.73	0.61	0.06

Table 4.2.4.2.2 Size at 90% Finer, D_{90} , μm

Material: Silicon Nitride, Test Powder
 Property: D_{90}
 Method: 13

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
8	35	1.36	1.57	1.45	0.05
9	54	1.18	1.49	1.32	0.06
14	4	1.25	1.60	1.42	0.15
20	3	1.09	1.11	1.10	0.01
22	30	0.99	1.32	1.13	0.09
24	4	1.51	2.01	1.79	0.22
25	3	0.99	1.01	1.00	0.01
Means	7	1.00	1.79	1.31	0.26

Table 4.2.4.2.3 Size at 10% Finer, D_{10} , μm

Material: Silicon Nitride, Test Powder
 Property: D_{10}
 Method: 13

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
8	35	0.35	0.37	0.36	0.00
9	54	0.28	0.32	0.30	0.00
14	4	0.17	0.23	0.21	0.02
20	3	0.32	0.32	0.32	0.00
22	30	0.30	0.32	0.30	0.00
24	4	0.32	0.41	0.35	0.03
25	3	0.30	0.30	0.30	0.00
Means	7	0.21	0.36	0.30	0.04

Table 4.2.4.2.4 Spread Ratio of Size Distribution, $(D_{90} - D_{10})/D_{50}$

Material: Silicon Nitride, Test Powder
 Property: $(D_{90} - D_{10})/D_{50}$
 Method: 13

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
8	35	1.54	1.67	1.60	0.03
9	54	1.47	1.92	1.67	0.09
14	4	1.79	2.51	2.23	0.34
20	3	1.33	1.36	1.34	0.01
22	30	1.18	1.67	1.44	0.13
24	4	1.61	2.32	1.96	0.36
25	3	1.25	1.30	1.28	0.02
Means	7	1.28	2.23	1.64	0.34

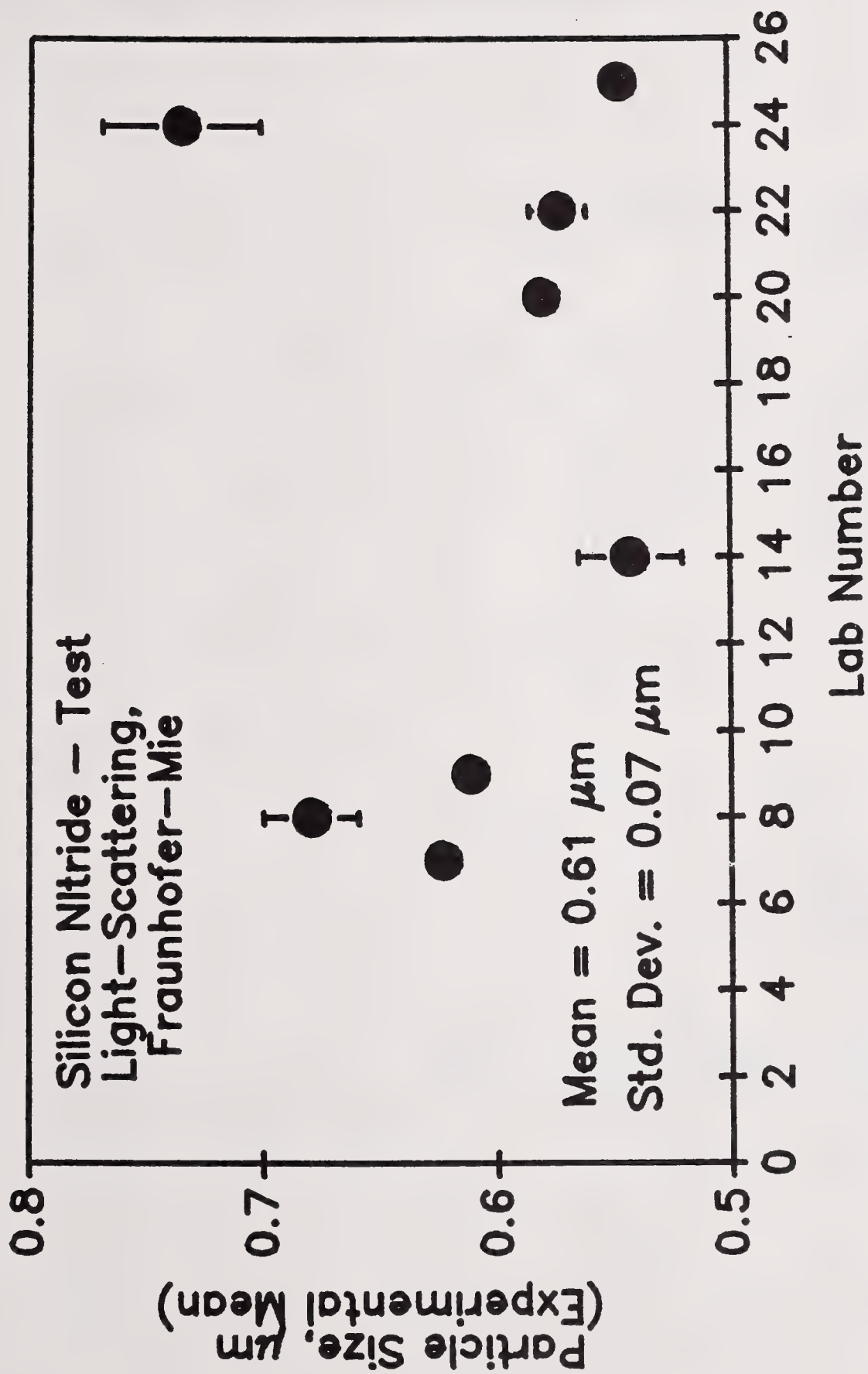


Figure 36. Experimental mean particle size of SNT powder by light-scattering, Fraunhofer-Mie type instrument.

4.2.4. Light Scattering Fraunhofer-Mie Type Instrument

4.2.4.3 Silicon Carbide Powder

Table 4.2.4.3.1 Experimental Mean, D_{50} , μm

Material: Silicon Carbide (Figure 37)
 Property: EXPTLMEAN
 Method: 13

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
7	3	0.39	0.44	0.42	0.02
8	24	0.50	0.52	0.51	0.00
10	4	0.48	0.50	0.48	0.00
20	1	0.72	0.72	0.72	0.00
21	1	0.57	0.57	0.57	0.00
22	47	0.55	0.66	0.59	0.02
25	1	0.53	0.53	0.53	0.00
Means	7	0.42	0.72	0.54	0.09

Table 4.2.4.3.2 Size at 90% Finer, D_{90} , μm

Material: Silicon Carbide
 Property: D_{90}
 Method: 13

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
7	3	0.64	0.67	0.66	0.01
8	24	0.98	1.01	0.98	0.00
10	4	0.99	1.52	1.19	0.24
20	1	1.61	1.61	1.61	0.00
22	47	0.91	1.64	1.22	0.15
25	1	1.17	1.17	1.17	0.00
Means	6	0.66	1.61	1.14	0.31

Table 4.2.4.3.3 Size at 10% Finer, D_{10} , μm

Material: Silicon Carbide
Property: D_{10}
Method: 13

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
7	3	0.23	0.29	0.27	0.03
8	24	0.29	0.30	0.29	0.00
10	4	0.23	0.23	0.23	0.00
20	1	0.35	0.35	0.35	0.00
22	47	0.29	0.36	0.32	0.01
25	1	0.29	0.29	0.29	0.00
Means	6	0.23	0.35	0.29	0.04

Table 4.2.4.3.4 Spread Ratio of Size Distribution, $(D_{90} - D_{10})/D_{50}$

Material: Silicon Carbide
Property: $(D_{90} - D_{10})/D_{50}$
Method: 13

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
7	3	0.86	1.05	0.92	0.10
8	24	1.33	1.40	1.34	0.01
10	4	1.55	2.58	1.97	0.46
20	1	1.75	1.75	1.75	0.00
22	47	1.02	2.15	1.50	0.23
25	1	1.66	1.66	1.66	0.00
Means	6	0.92	1.97	1.52	0.36

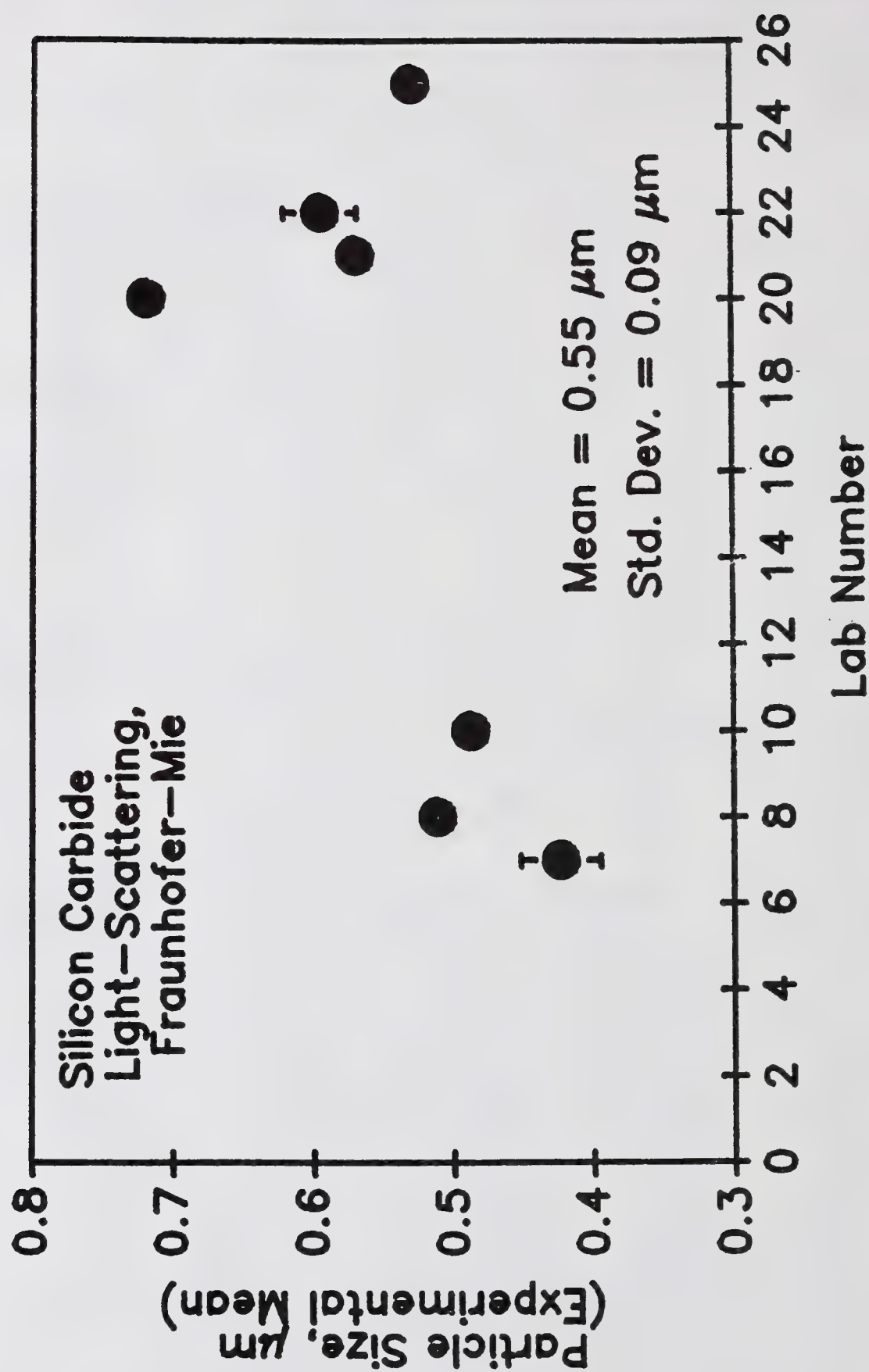


Figure 37. Experimental mean particle size of SiC powder by light-scattering, Fraunhofer-Mie type instrument.

4.2.4. Light-Scattering Fraunhofer-Mie Type Instruments

4.2.4.4 Silicon Powder

Table 4.2.4.4.1 Experimental Mean, D_{50} , μm

Material: Silicon (Figure 38)
 Property: EXPTLMEAN
 Method: 13

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
5	2	6.50	6.50	6.50	0.00
8	24	7.12	7.78	7.37	0.15
9	36	7.34	7.61	7.43	0.06
20	1	7.36	7.36	7.36	0.00
22	24	7.09	8.10	7.57	0.27
Means	5	6.50	7.57	7.25	0.42

Table 4.2.4.4.2 Size at 90% Finer, D_{90} , μm

Material: Silicon
 Property: D_{90}
 Method: 13

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
5	2	11.70	11.80	11.75	0.07
8	24	12.84	15.01	13.95	0.48
9	36	13.60	14.39	13.98	0.19
20	1	13.89	13.89	13.89	0.00
22	24	12.61	15.14	13.80	0.69
Means	5	11.75	13.98	13.47	0.96

Table 4.2.4.4.3 Size at 10% Finer, D_{10} , μm

Material: Silicon
Property: D_{10}
Method: 13

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
5	2	1.60	1.60	1.60	0.00
8	24	2.40	2.81	2.58	0.10
9	36	2.73	3.02	2.88	0.06
20	1	2.70	2.70	2.70	0.00
22	24	2.06	2.94	2.35	0.24
Means	5	1.60	2.88	2.42	0.49

Table 4.2.4.4.4 Spread Ratio of Size Distribution, $(D_{90} - D_{10})/D_{50}$

Material: Silicon
Property: $(D_{90} - D_{10})/D_{50}$
Method: 13

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
5	2	1.55	1.57	1.56	0.01
8	24	1.42	1.64	1.54	0.05
9	36	1.45	1.54	1.49	0.02
20	1	1.52	1.52	1.52	0.00
22	24	1.31	1.66	1.51	0.09
Means	5	1.49	1.56	1.52	0.02

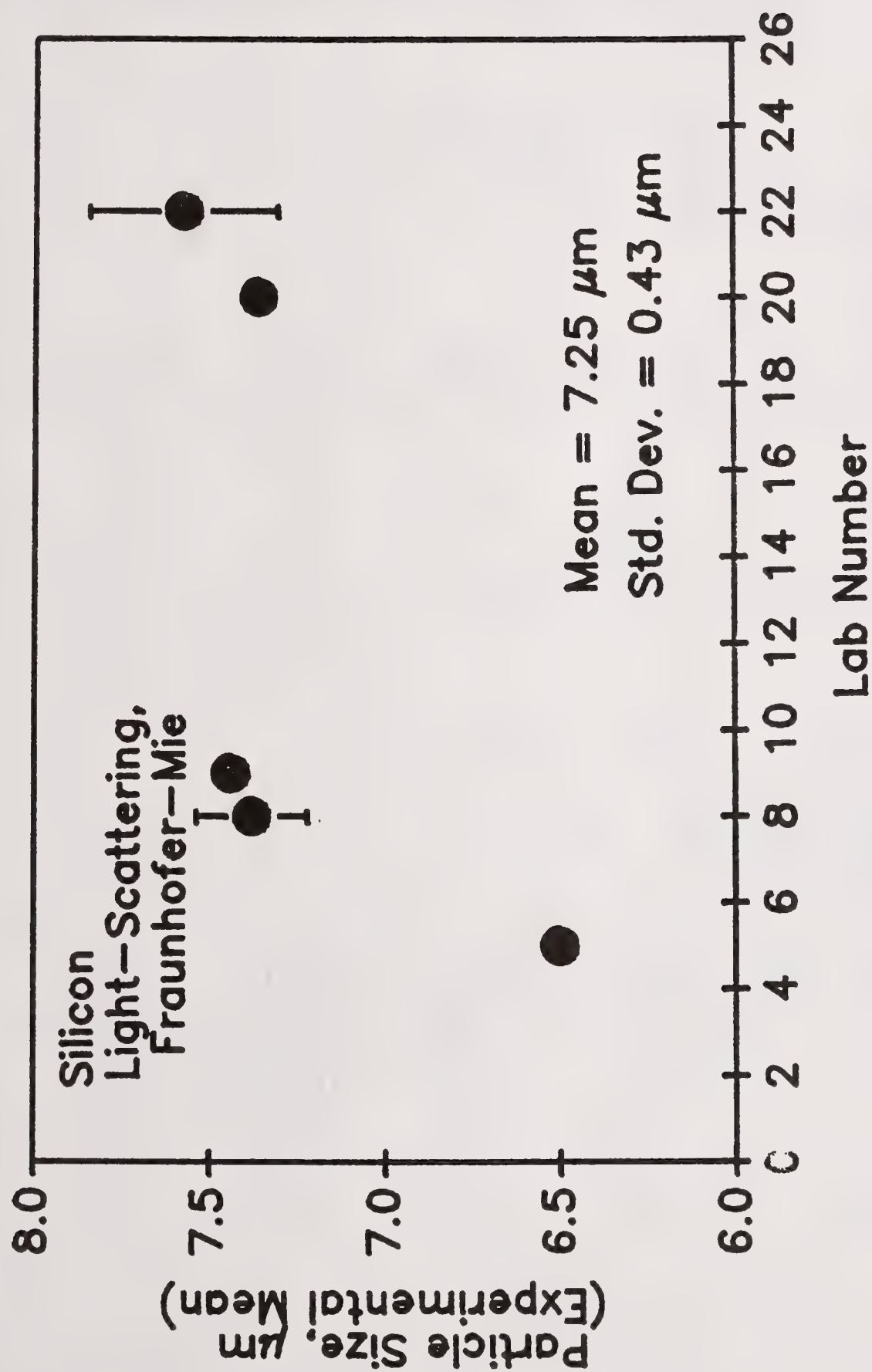


Figure 38. Experimental mean particle size of Si powder by light-scattering, Fraunhofer-Mie type instrument.

4.2.4. Light Scattering Fraunhofer-Mie Type Instruments

4.2.4.5 Yttria-Zirconia Powder

Table 4.2.4.5.1 Experimental Mean, D_{50} , μm

Material: Zirconia (Figure 39)
 Property: EXPTLMEAN
 Method: 13

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
11	1	2.90	2.90	2.90	0.00
14	3	2.20	2.30	2.26	0.05
20	3	4.06	4.19	4.14	0.07
22	7	0.62	0.95	0.76	0.11
24	3	1.40	1.80	1.56	0.20
Means	5	0.76	4.14	2.32	1.28

Table 4.2.4.5.2 Size at 90% Finer, D_{90} , μm

Material: Zirconia
 Property: D_{90}
 Method: 13

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
11	1	9.20	9.20	9.20	0.00
14	3	9.50	9.80	9.60	0.17
20	3	11.33	11.80	11.54	0.23
22	7	2.38	2.96	2.65	0.20
24	3	7.60	8.60	7.96	0.55
Means	5	2.65	11.54	8.19	3.35

Table 4.2.4.5.3 Size at 10% Finer, D_{10} , μm

Material: Zirconia
 Property: D_{10}
 Method: 13

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
11	1	0.37	0.37	0.37	0.00
14	3	0.40	0.40	0.40	0.00
20	3	0.50	0.51	0.50	0.00
22	7	0.15	0.15	0.15	0.00
24	3	0.13	0.14	0.13	0.00
Means	5	0.13	0.50	0.31	0.16

Table 4.2.4.5.4 Spread Ratio of Size Distribution, $(D_{90} - D_{10})/D_{50}$

Material: Zirconia
 Property: $(D_{90} - D_{10})/D_{50}$
 Method: 13

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
11	1	3.04	3.04	3.04	0.00
14	3	3.96	4.14	4.06	0.09
20	3	2.62	2.71	2.66	0.04
22	7	2.71	4.12	3.31	0.55
24	3	4.70	5.34	5.03	0.32
Means	5	2.66	5.03	3.62	0.93

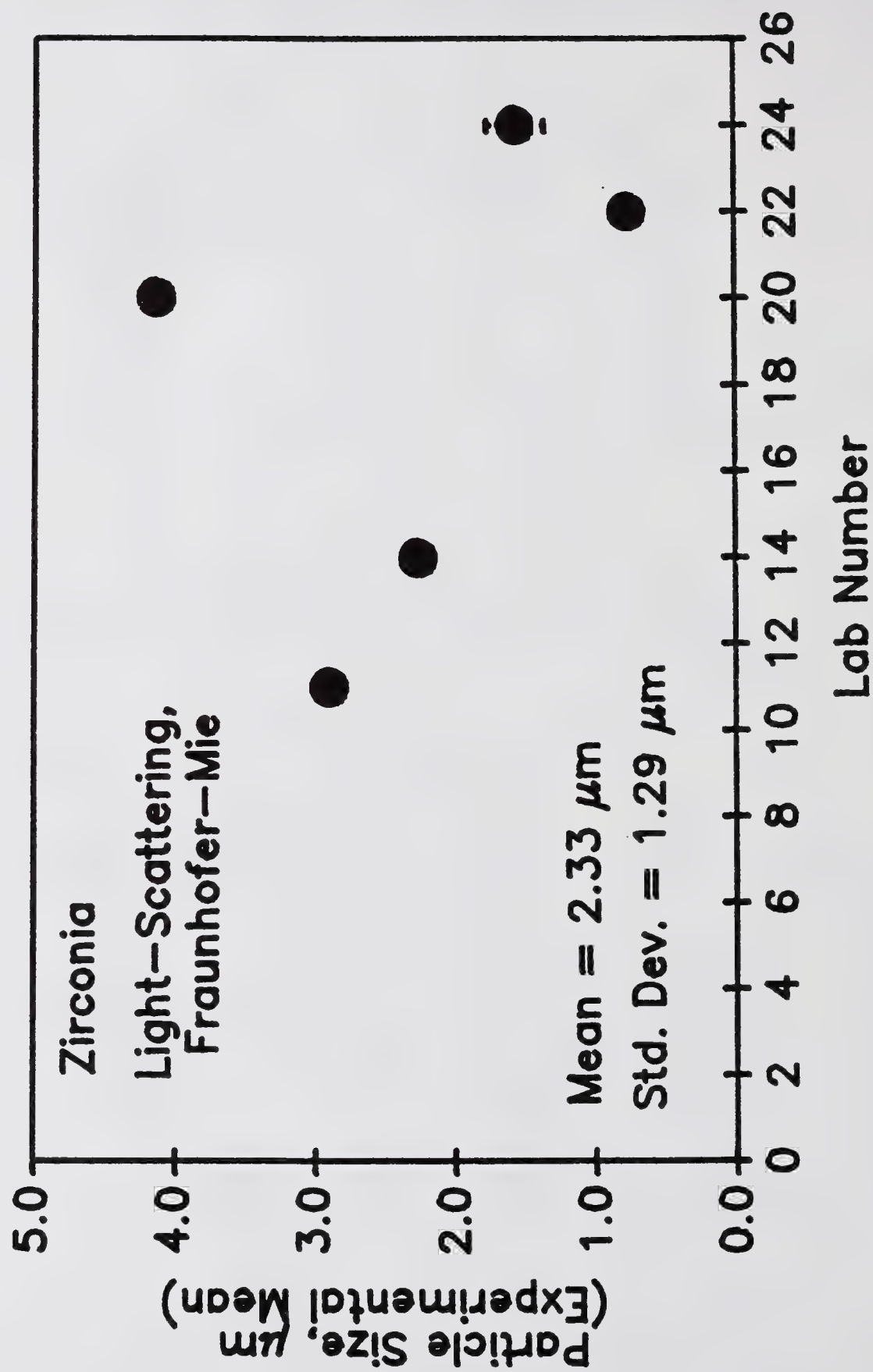


Figure 39. Experimental mean particle size of YSZ powder by light-scattering, Fraunhofer-Mie type instrument.

4.2.5. Electrical Sensing Zone

4.2.5.1 Silicon Powder

Table 4.2.5.1.1 Experimental Mean, D_{50} , μm

Material: Silicon
Property: EXPTLMEAN
Method: 7

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
16	6	5.20	5.50	5.31	0.11
Means	1	5.31	5.31	5.31	0.00

Table 4.2.5.1.2 Size at 90% Finer, D_{90} , μm

Material: Silicon
Property: D_{90}
Method: 7

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
16	5	9.60	10.00	9.82	0.20
Means	1	9.82	9.82	9.82	0.00

Table 4.2.5.1.3 Size at 10% Finer, D_{10} , μm

Material: Silicon
Property: D_{10}
Method: 7

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
16	5	1.05	1.15	1.08	0.03
Means	1	1.08	1.08	1.08	0.00

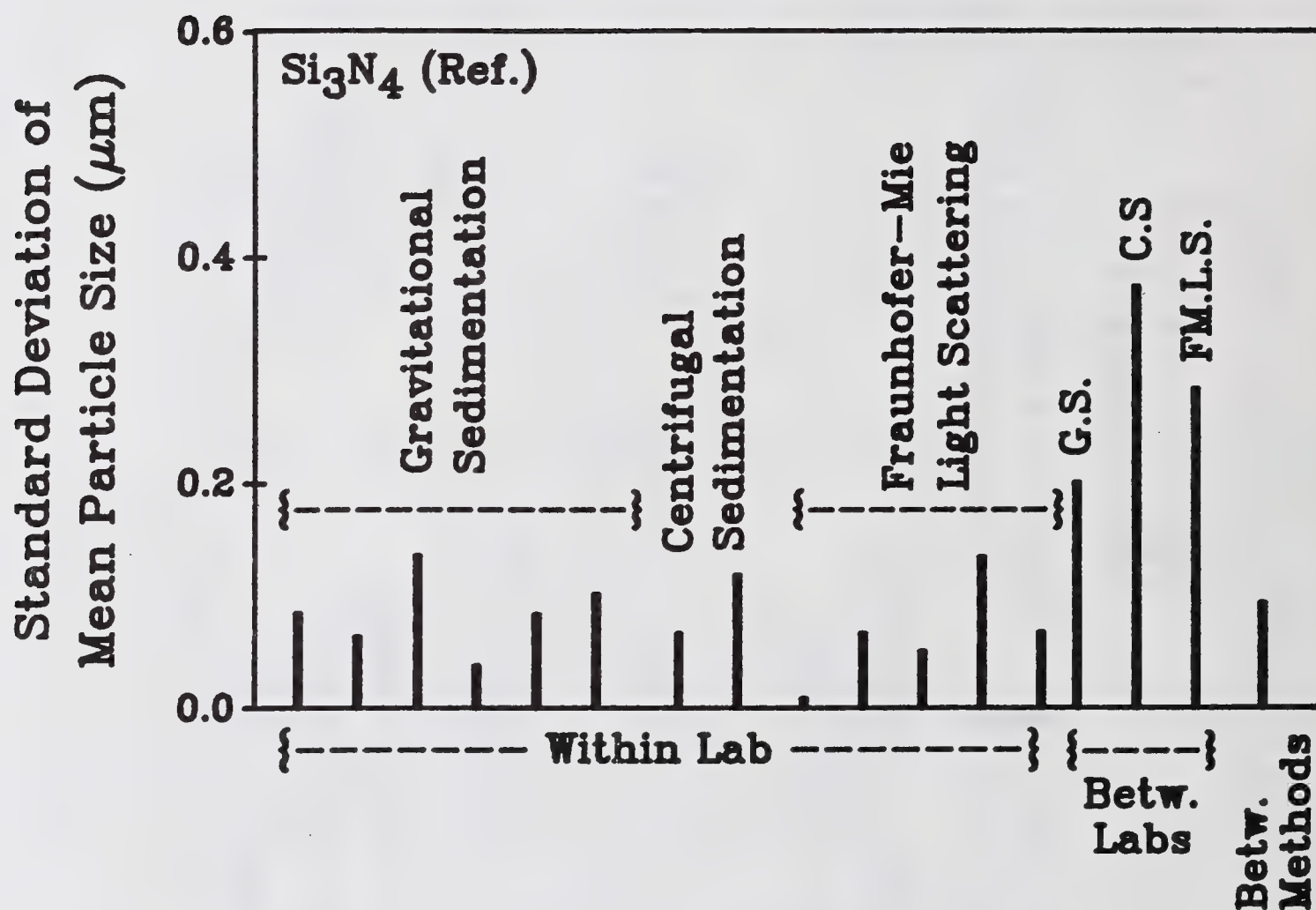


Figure 40.

Within lab, between lab, and between methods standard deviations in the measurement of particle size for the Si_3N_4 (Ref.) powder as measured by various laboratories using a variety of methods.

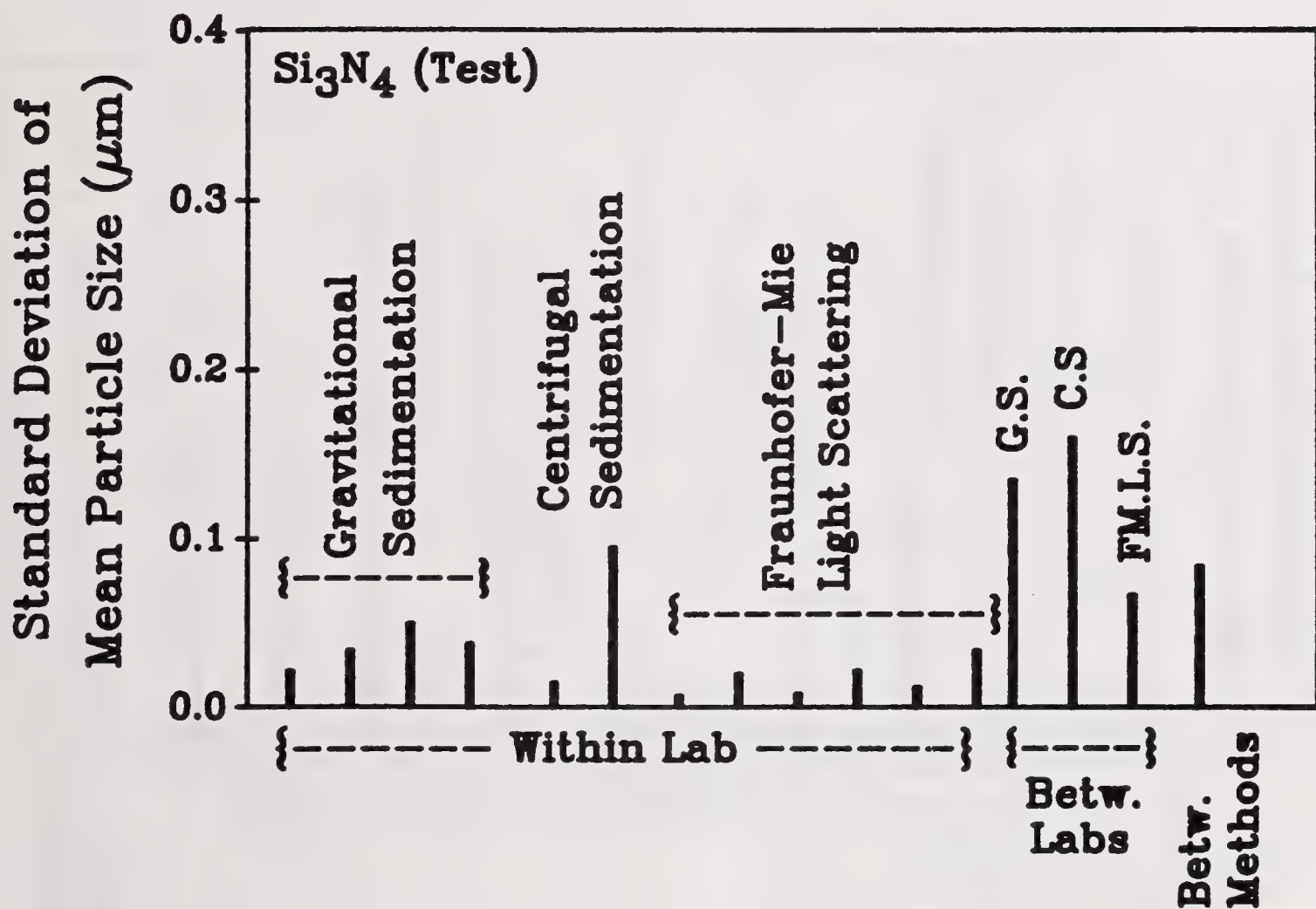


Figure 41.

Within lab, between lab, and between methods standard deviations in the measurement of particle size for the Si₃N₄(Test) powder as measured by various laboratories using a variety of methods.

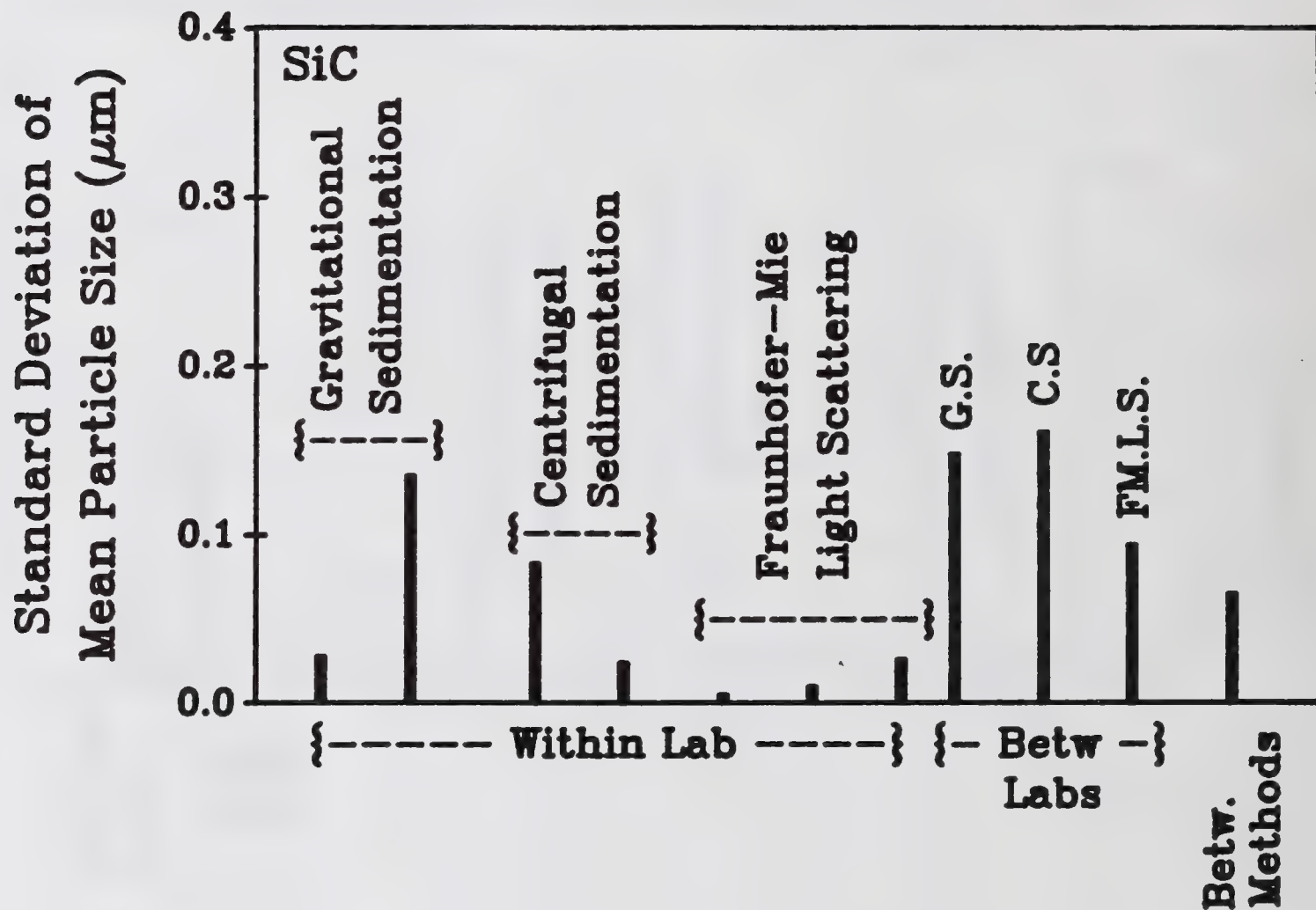


Figure 42.

Within lab, between lab, and between methods standard deviations in the measurement of particle size for the SiC powder as measured by various laboratories using a variety of methods.

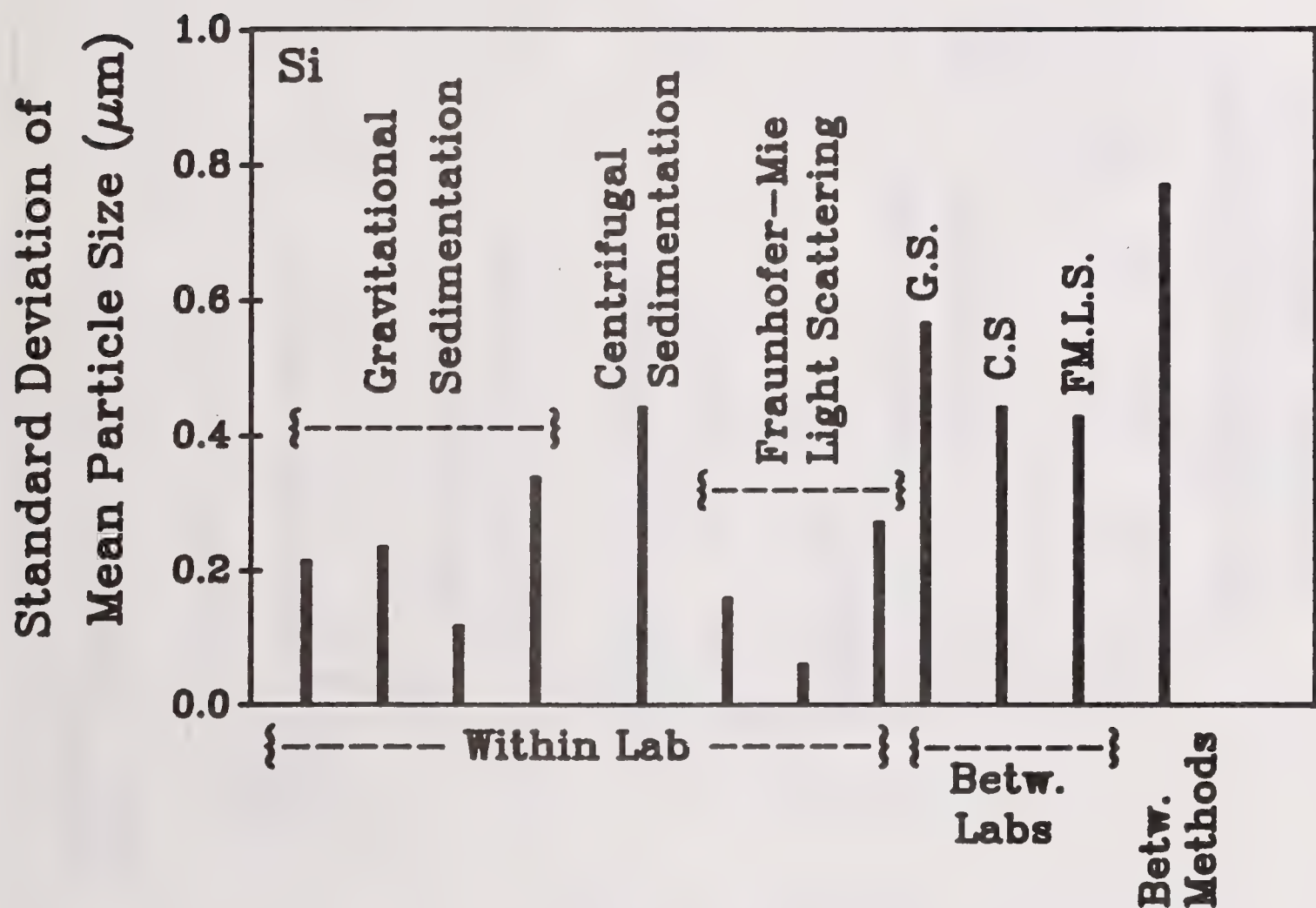


Figure 43.

Within lab, between lab, and between methods standard deviations in the measurement of particle size for the Si powder as measured by various laboratories using a variety of methods.

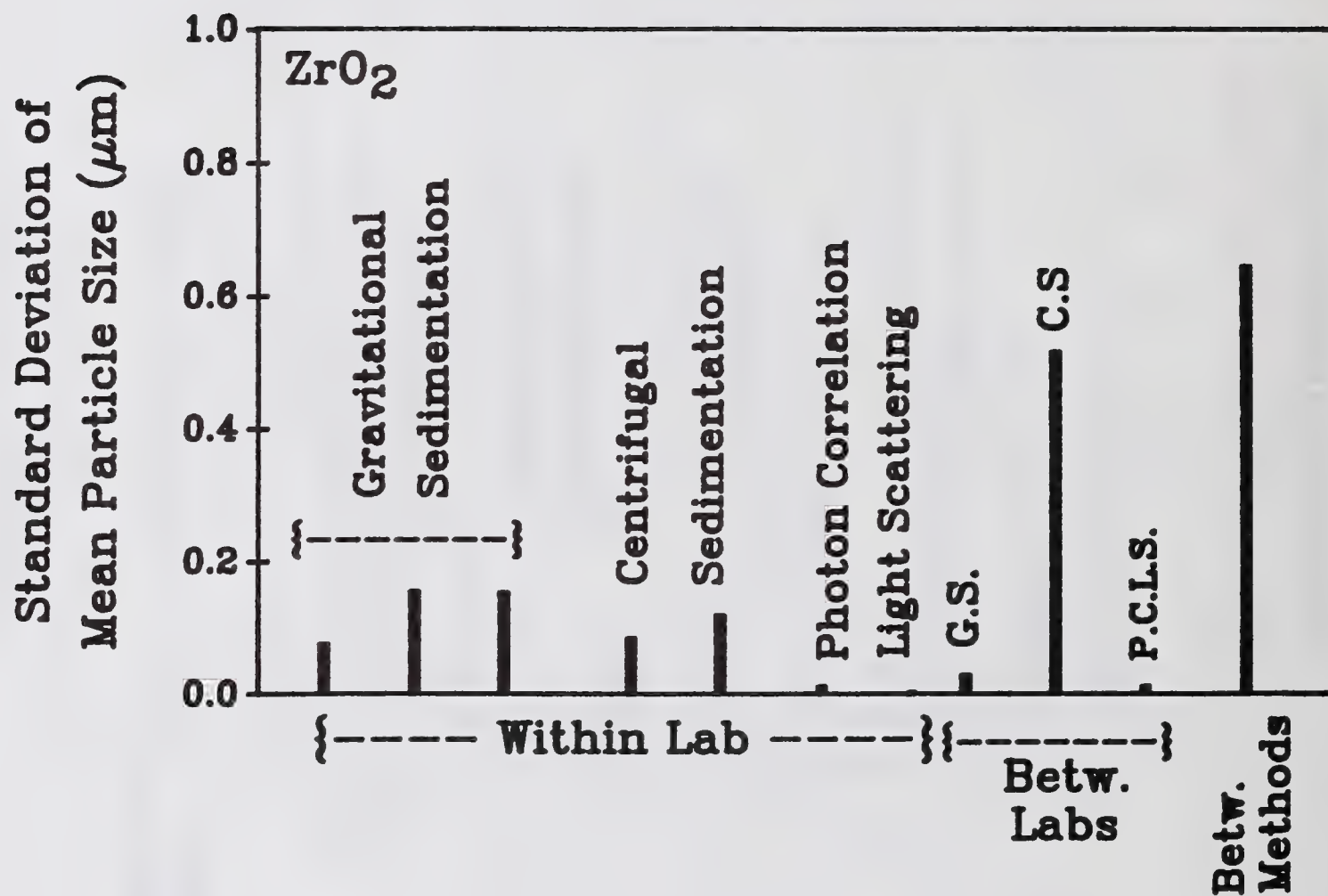


Figure 44.

Within lab, between lab, and between methods standard deviations in the measurement of particle size for the ZrO₂ powder as measured by various laboratories using a variety of methods.

Table 4.2.5.1.4 Spread Ratio of Size Distribution, $(D_{90} - D_{10})/D_{50}$

Material: Silicon
 Property: $(D_{90} - D_{10})/D_{50}$
 Method: 7

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
16	5	1.61	1.66	1.64	0.01
Means	1	1.64	1.64	1.64	0.00

4.3. STATISTICAL ANALYSIS, R. Munro, NIST

Several techniques are available to measure particle size distributions in powders. These techniques may be based on different physical principles and different instrumentation and may utilize significantly different procedures for dispersing the particles in a medium before a measurement is performed. Further, the various methods have different detection limits.

Two of the methods, gravitational sedimentation and centrifugal sedimentation, utilize the size dependence of the terminal velocity of particles moving through a viscous medium under the influence of gravity or the rotational acceleration of the medium. In both methods, an equivalent spherical diameter is assumed as the measure of the particle size. Two other methods, Fraunhofer-Mie light scattering and photon correlation spectroscopy, depend on the size dependence of the distribution or diffraction pattern of the scattered light. These techniques determine a mean size of the particles in the diffraction volume.

Results from gravitational sedimentation and centrifugal sedimentation are shown in Figures 40-44; results for Fraunhofer-Mie light scattering are shown in Figures 40-43 for all powders except zirconia; and results for photon correlation spectroscopy are shown in Figure 44 for zirconia. Each method measures the cumulative particle size distribution and computes the mean particle size from that distribution. In each case, the standard deviation was typically less than ten percent of the mean value, and none of the methods was consistently better than the others. The variances between laboratories were larger than the differences between methods for silicon nitride and silicon carbide, but for silicon and zirconia, the discrepancy between methods was larger.

5. PARTICLE MORPHOLOGY

R. Pompe, Swedish Ceramic Institute; A. L. Dragoo, NIST

5.1. MEASUREMENT METHODS

5.1.1. Scanning Electron Microscopy (SEM)/Transmission Electron Microscopy (TEM)

Use and Significance

Microscopic methods of particle evaluation (SEM, TEM) provide a direct means of estimating the particle size distribution, degree of agglomeration, particle or agglomerate shape and aspect ratio. Both techniques are used extensively to obtain images, usually as micrographs of particles, which provide a picture of particle features and which offer a qualitative, order-of-magnitude estimate of particle size. By coupling SEM and TEM with quantitative image analysis, quantitative or semiquantitative evaluation of particle size and shape characteristics can be obtained. A wide size range down to approximately 2 nm can be covered with a combination of the two techniques. Powder sample preparation techniques are crucial in obtaining a representative image. By virtue of being the only methods of direct particle observation, they are essential for powder characterization in conjunction with other physical methods.

Instrument and Experimental Parameters

SEM/TEM micrographs were supplied by the following laboratories:

Lab #	Powder	Mode
-----	-----	-----
4	All Powders	SEM
18	SNT, SiC, Si, YSZ	SEM
22	YSZ	TEM
23	SNR	SEM

Labs 12, 13, 18 and 23 conducted quantitative image analysis of various powders. The results of the image analysis are summarized in the subsequent sections.

Sample Preparation

For qualitative studies, powder samples were deposited on SEM sample holders in the as-received state or dispersed and deposited dropwise on a grid. The following is an example of preparation procedure used by Lab 6:

SiC Powder

As-received-

A small amount of powder was sprinkled on a sticky tab which was placed on a carbon grid. The excess was removed and a thin layer of Au-Pd metallization was applied to the sample. Photomicrographs were taken at 200x, 500x, 1000x and 2000x.

Dispersed-

Approximately 3 mg of powder was placed in a beaker which contained 80 ml of distilled water to which had been added 16 drops of concentrated ammonium hydroxide (pH $\approx$ 10.5). The sample was dispersed for 30 s in a sonifier. Several drops of the dispersed sample were placed on a carbon grid and allowed to dry. A thin layer of Au-Pd metallization was applied to the samples. Photomicrographs were taken at 2000x, 5000x and 7500x.

Lab 4 prepared samples by the camphor-naphthalene method. A mixture of 10 mg powder and 4 mg camphor-naphthalene (6:4) was melted in an oven and was then ultrasonicated for 4 minutes. After rapid cooling to -20 °C a small amount of the mixture was suspended on to a preheated SEM aluminum stub and the organic part was sublimed away.

For quantitative analysis of particle size, Lab 23 reported the dispersion of a few milligrams of powder in 25 ml of water which contained 0.02 % Darvan. The suspension was ultrasonicated for 60 s, then filtered through a polycarbonate membrane filter with 0.2 μ m diameter pores. The filter and particles were coated with 5-10 nm of Au-Pd.

Calibration and Standards

In addition to many reference artifacts and resolution grids frequently supplied by manufacturers, there are several certified references available from the National Institute of Standards and Technology; namely,

SRM 484e	SEM Magnification Standard
SRM 2069a	SEM Performance Standard.

In addition, SRM's 1690, 1691, 1960, 1961 and 1965, which consist of calibrated polystyrene spheres in water can be incorporated into particle suspensions and applied to SEM grids as a length reference.

Results and Discussion

Qualitative Analysis

Micrographs illustrating various aspects of the powders are presented in Section 5.2.

The main features of all the data received of dispersed powders are summarized and the relevant SEM/TEM micrographs are collected in Sections 5.2 and 5.3.

Quantitative Analysis

Data for particle size analysis are summarized in Section 5.3.

Lab 23 reported results for all powders. Measurements were made on approximately 2000 particles for each powder, using backscattered electrons. The video output of the SEM was processed by a Kevex Delta V image analyzer. The particle dimension was given in terms of the Wadell diameter (i.e., the diameter of a circle having the same area as that of the image of a particle). Five to seven size intervals were used to obtain approximate representations of the size distributions for each powder. Number-based distributions were transformed to mass(volume)-based distributions by assuming equivalent spherical volumes for the particles.

Lab 23 reported determinations of the average particle size for SNR samples; see Table 5.3.2.1. A 30-40 mg portion of each powder was added to 100 ml of a surfactant solution and dispersed ultrasonically for 5 minutes. One drop of the suspension was air dried on a glass slide and then coated with carbon and Au-Pd conductive coating. Image data were collected from calibrated micrographs. Results were reported in terms of the arithmetic mean of the maximum length of the observed grains.

Lab 12 reported one average particle size for each of samples SNR, SNT, and Si powders, and two results for the YSZ powder. The values reported by Lab 12 appear to be those for mass-based distributions, but this could not be confirmed.

Lab 18 analyzed as-received and dispersed samples of the YSZ powder. Particle images on the SEM micrographs were examined with a Zeiss Interactive Digital System by tracing the images and quantitatively determining agglomerate sizes and the size distribution. The results of the analysis are summarized in Table 5.1.1.1 below and, for six samples of the as-received powder, in Table 5.3.6.1. For the quantitative analysis of the as-received powder, between 109 and 305 particles were measured per sample. The mean size and the standard deviation (not σ_g) were reported for each powder. They reported the number of particles versus size, for IEA powder OR012, using a total of 22 intervals. Since the spray-dried agglomerates of the as-received powder were approximately spherical in shape, a mass-based distribution can be calculated with a fair degree of accuracy from the number-based distribution which they reported. These two distributions are shown in Figure 45 and illustrate the marked difference which exists between the two distributions.

Table 5.1.1.1 Summary of Agglomerate and Particle Characteristics for
Yttria-Zirconia (YSZ) Powder (Lab 18)

1. Primary Agglomerates (As-received Powder)

Determination:	SEM
Average Size:	30-35 μm (Number)
Shape:	Spherical
% Agglomerated:	> 90%
Aspect Ratio:	1

2. Secondary Agglomerates (Dispersed Powder)

Determination:	SEM and Sedigraph
Average Size:	0.1-0.2 μm
Shape:	Equiaxed
Aspect Ratio:	Not determined

3. Crystallite Size

Determination:	XRD
Average Size:	22-23 nm

Conclusions

Qualitative SEM/TEM evaluation is an important complement to other particle characterization methods in terms of identifying the fabrication route and of gaining information on the shape, aspect ratio and agglomeration state of the particular powder.

Computer-automated techniques for image analysis can be utilized for measuring average particle size. Detailed quantitative information can be obtained, such as primary agglomerate size etc., which can be correlated to other, physical methods of particle size distribution measurements.

YTTRIA-ZIRCONIA POWDER AS RECEIVED

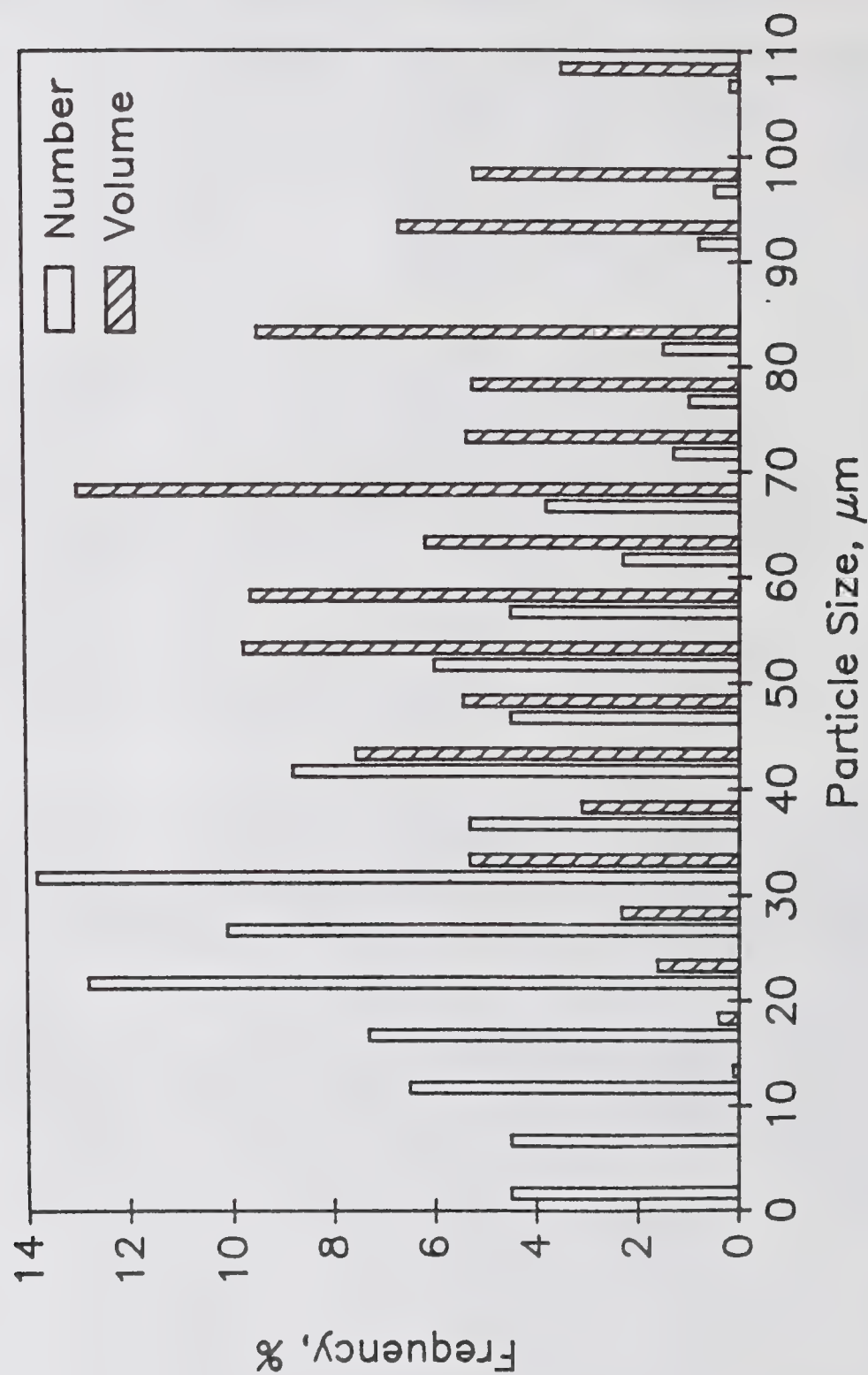


Figure 45. Number-based and mass(volume)-based particle size distributions for as-received yttria-zirconia (YSZ) powder.

5.2. PARTICLE MORPHOLOGY - MICROGRAPHS

5.2.1. Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM)

Particle morphology of as-received powders and of dispersed powders and particles was characterized by SEM and TEM studies. Selected micrographs of the five powders are presented in the following pages.

5.2.1.1 Silicon Nitride Reference Powder



Figure 46. SEM micrograph of powder as-received. Magnification 100x; scale bar in lower right of micrograph indicates 100 μm length. Sample: SNR 9028. Lab No. 4.

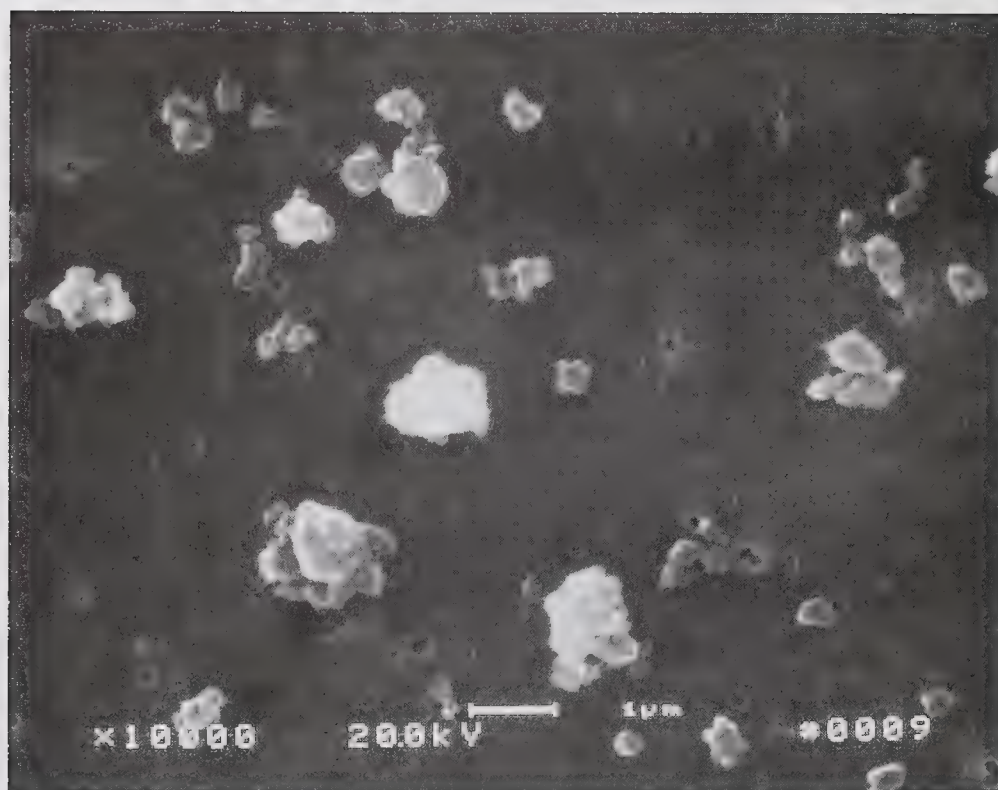


Figure 47.

SEM micrograph of powder after dispersal in water and followed by collection of polycarbonate filter. Scale bar in lower center of micrograph indicates 1 μm length. Sample: SNR 0408. Lab No. 23.



Figure 48.

SEM micrograph of particle collected on polycarbonate filter. Note dense agglomerated structure of particle. Scale bar in lower center of micrograph indicates 1 μm length. Sample: SNR 0408. Lab No. 23.



Figure 49. SEM micrograph of agglomerated SNR particles. Magnification 40,000x; scale bar in lower right of micrograph indicates 1 μm length. Sample: SNR 9028. Lab No. 4.

5.2.1.2.

Silicon Nitride Test Powder

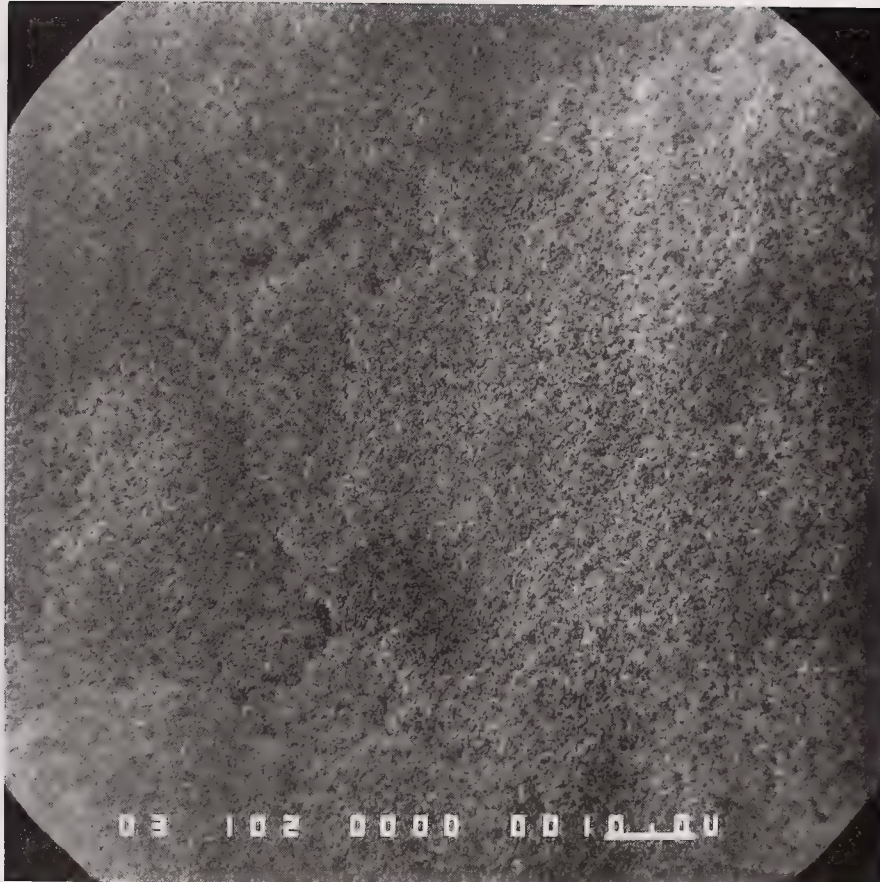


Figure 50.

SEM micrograph of powder as-received. Magnification 1000x; scale bar in lower right of micrograph indicates 10 μm length. Sample: SNT LSM 2. Lab No. 4.

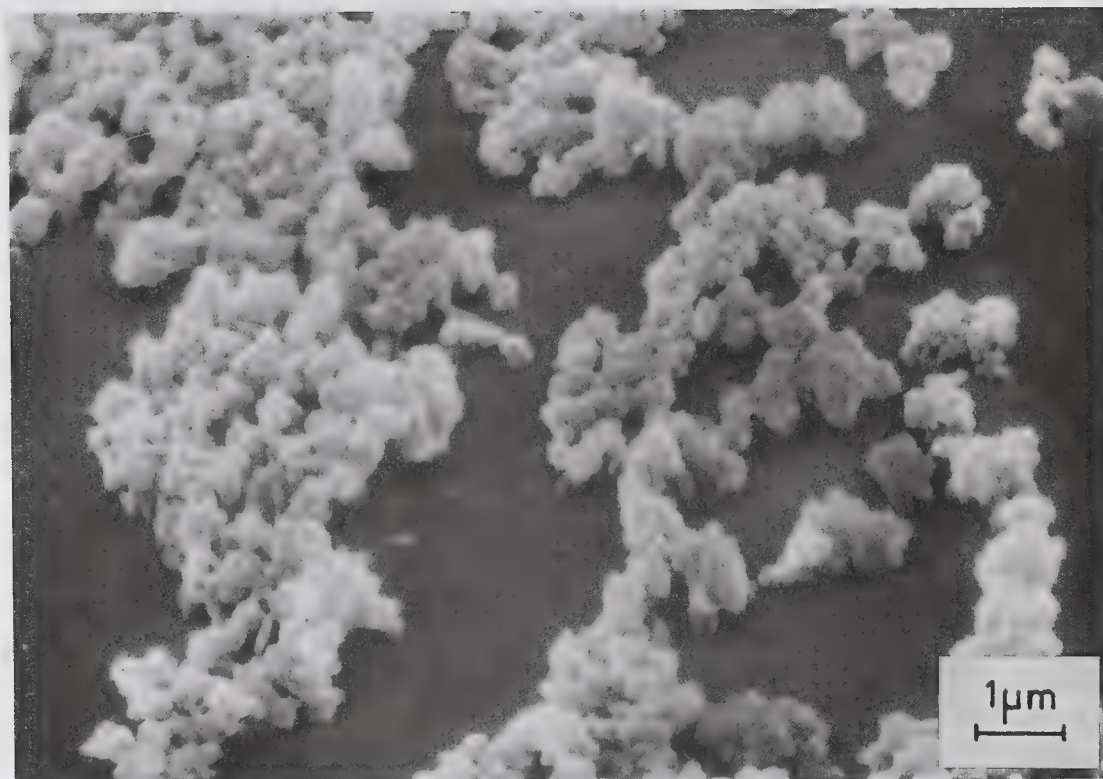


Figure 51. SEM micrograph of powder. Sample: SNT 1385. Lab No. 21.

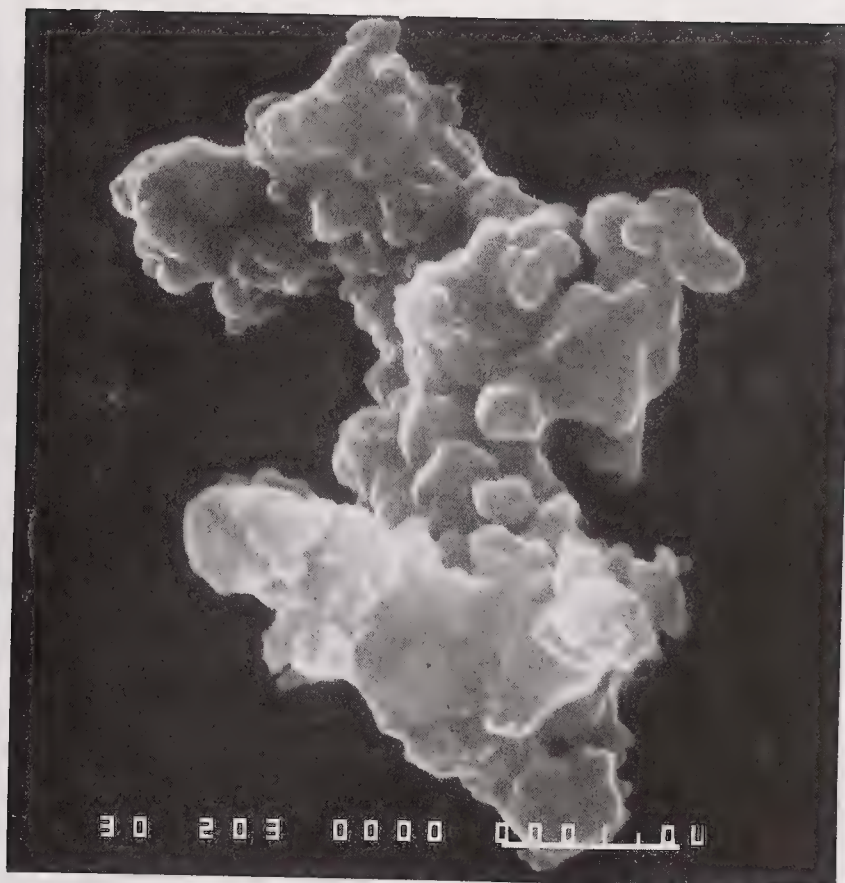


Figure 52. SEM micrograph of particle. Magnification 20,000x; scale bar in lower right of micrograph indicates 1 μm length. Sample: SNT LSM 2. Lab No. 4.

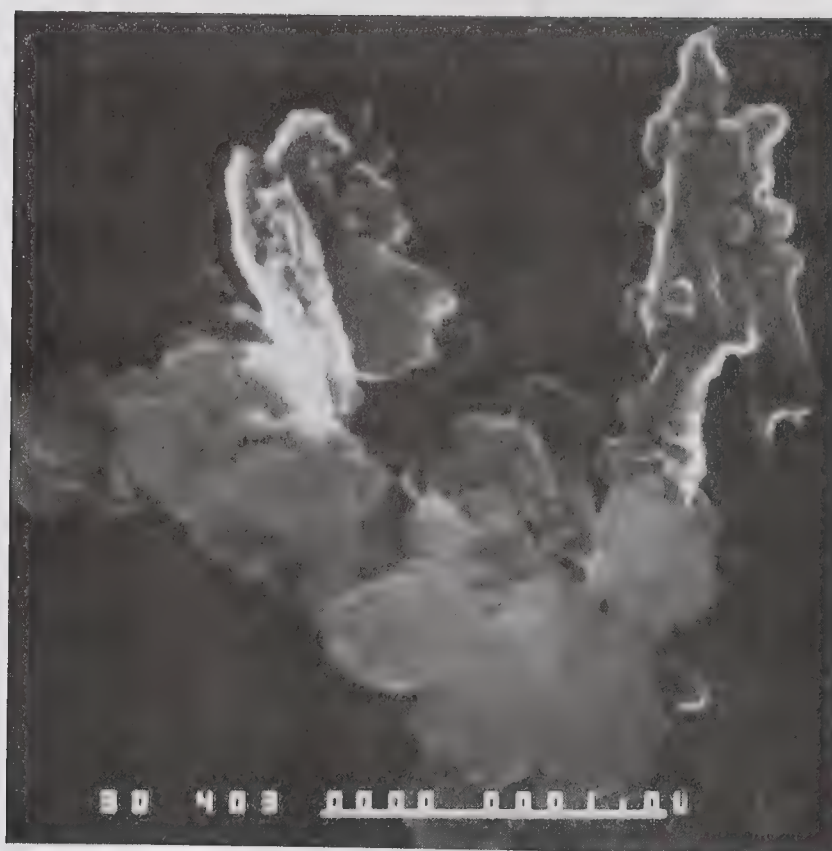


Figure 53. SEM micrograph of particle. Magnification 40,000x; scale bar in lower right of micrograph indicates 1 μm length. Sample: SNT LSM 2. Lab No. 4.

5.2.1.3.

Silicon Carbide Powder



Figure 54.

SEM micrograph of powder as-received. Magnification 1000x; scale bar in lower right of micrograph indicates 10 μm length. Sample: SiC 1023. Lab No. 4.

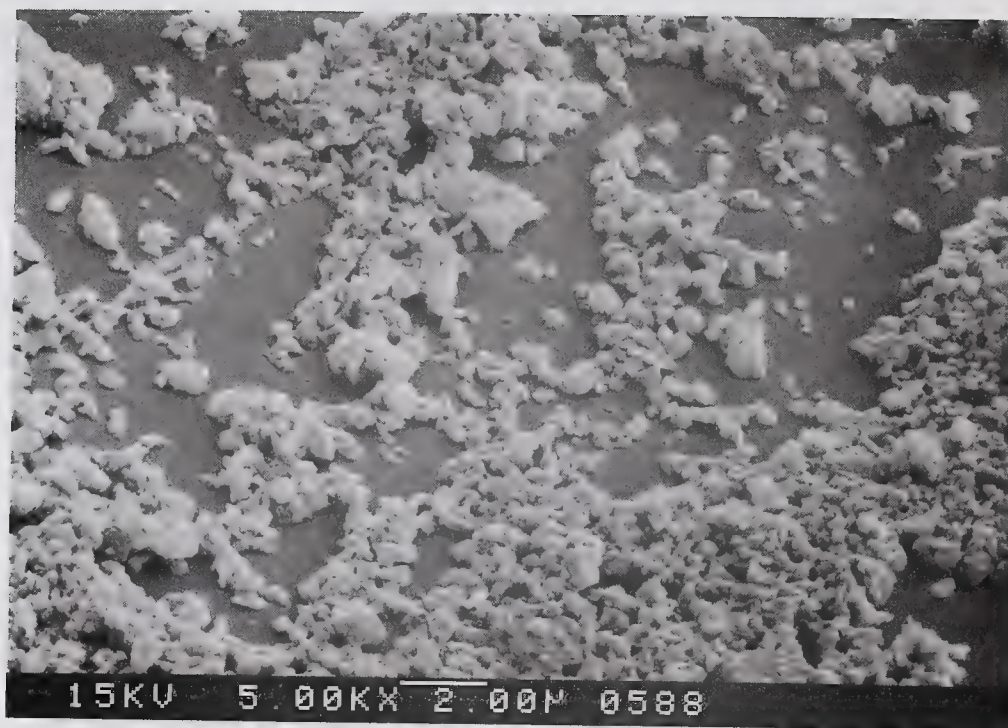


Figure 55. SEM micrograph of powder. Scale bar in lower center of micrograph indicates 2 μm length. Sample: SiC 0588. Lab No. 21.

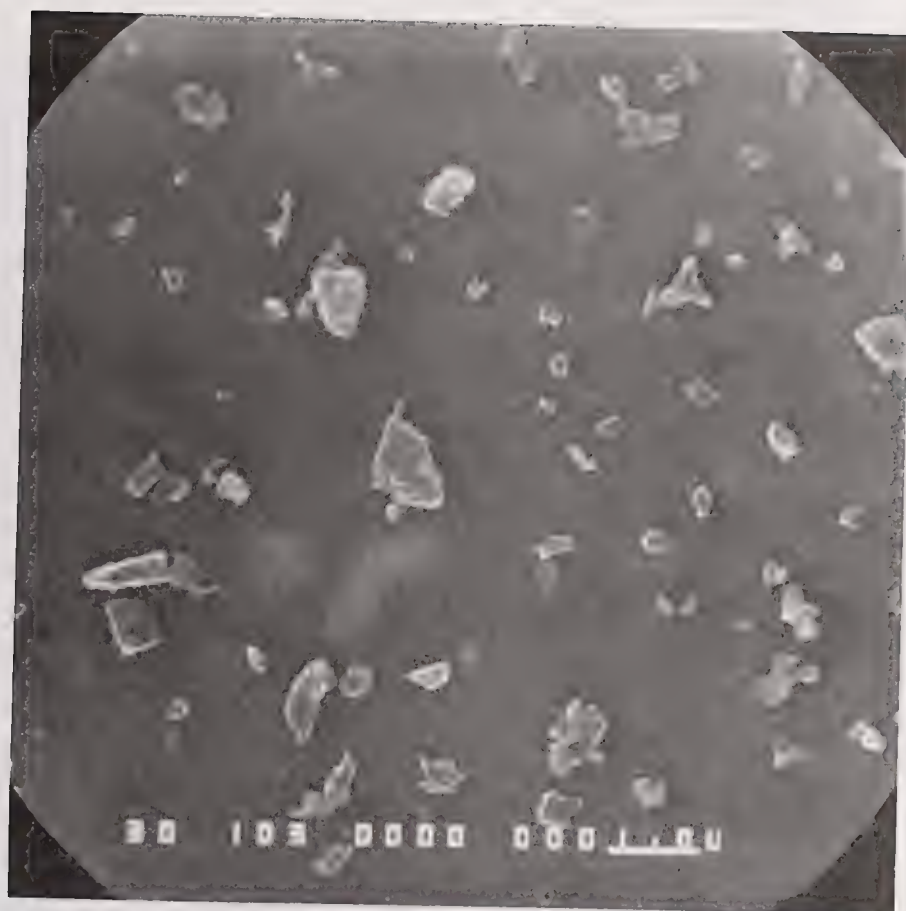


Figure 56. SEM micrograph of dispersed powder. Magnification 10,000x; scale bar in lower right of micrograph indicates 1 μm length. Sample: SiC 1023. Lab No. 4.

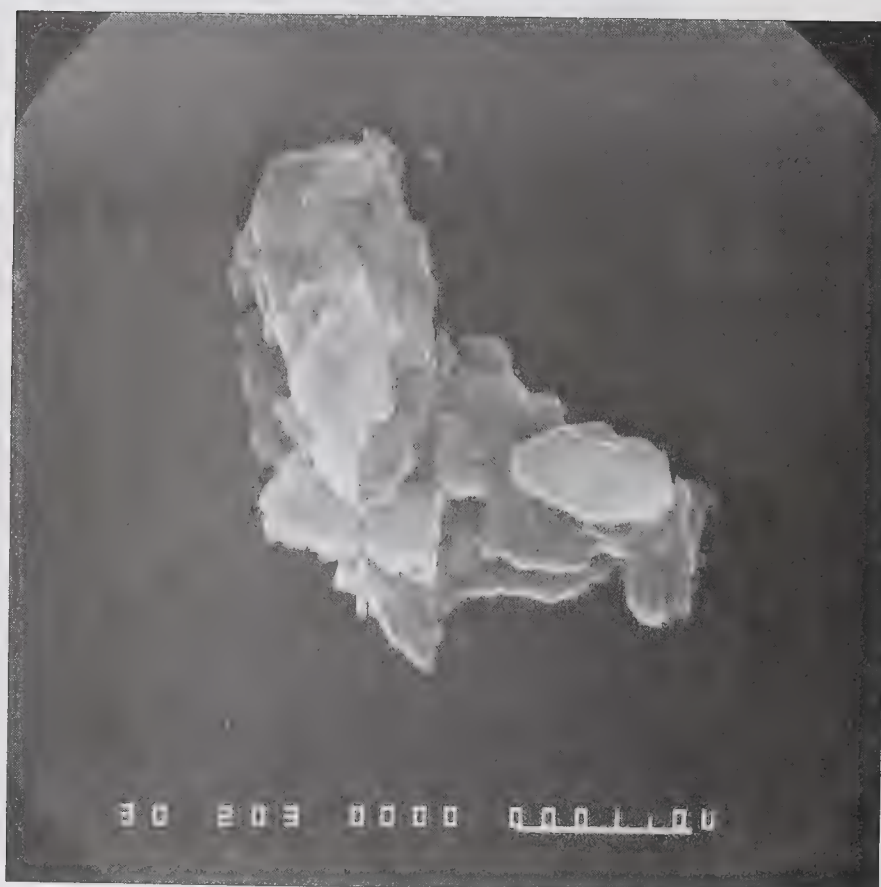


Figure 57. SEM micrograph of particle. Magnification 20,000x; scale bar in lower right of micrograph indicates 1 μm length. Sample: SiC 1023. Lab No. 4.

5.2.1.4.

Silicon Powder

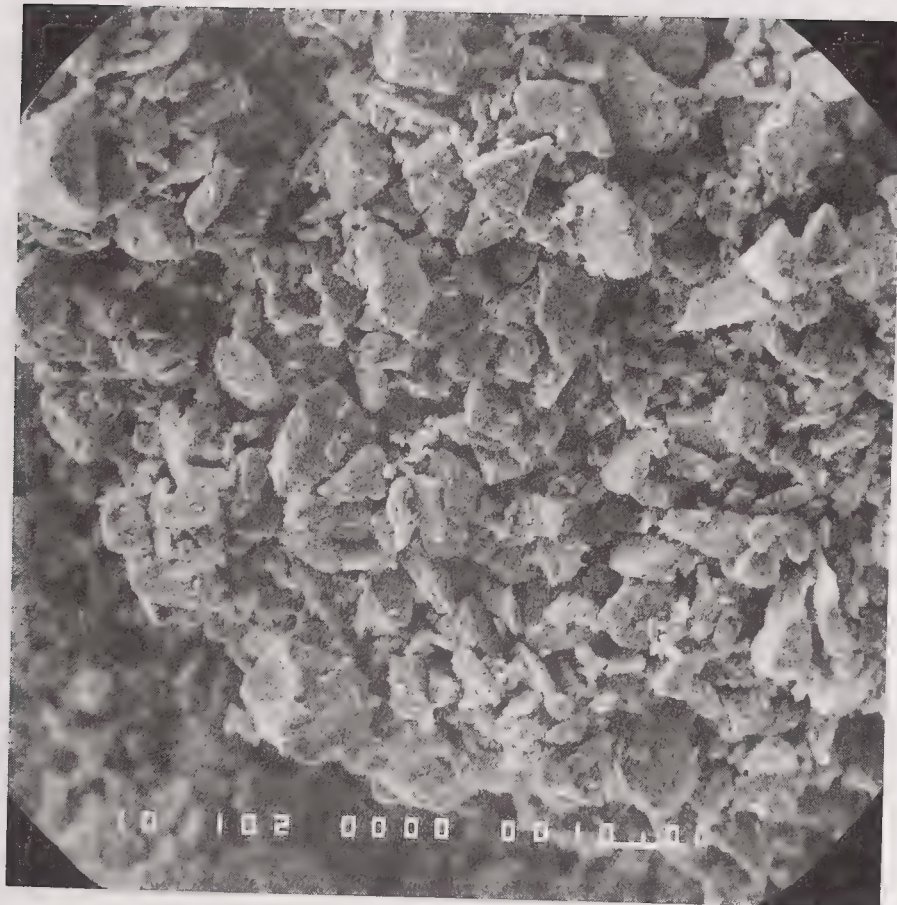


Figure 58.

SEM micrograph of powder as-received. Magnification 1000x; scale bar in lower right of micrograph indicates 10 μm length. Sample: Si 1001. Lab No. 4.



Figure 59.

SEM micrograph of dispersed powder. Magnification 1000x; scale bar in lower right of micrograph indicates 10 μm length. Sample: Si 1001. Lab No. 4.

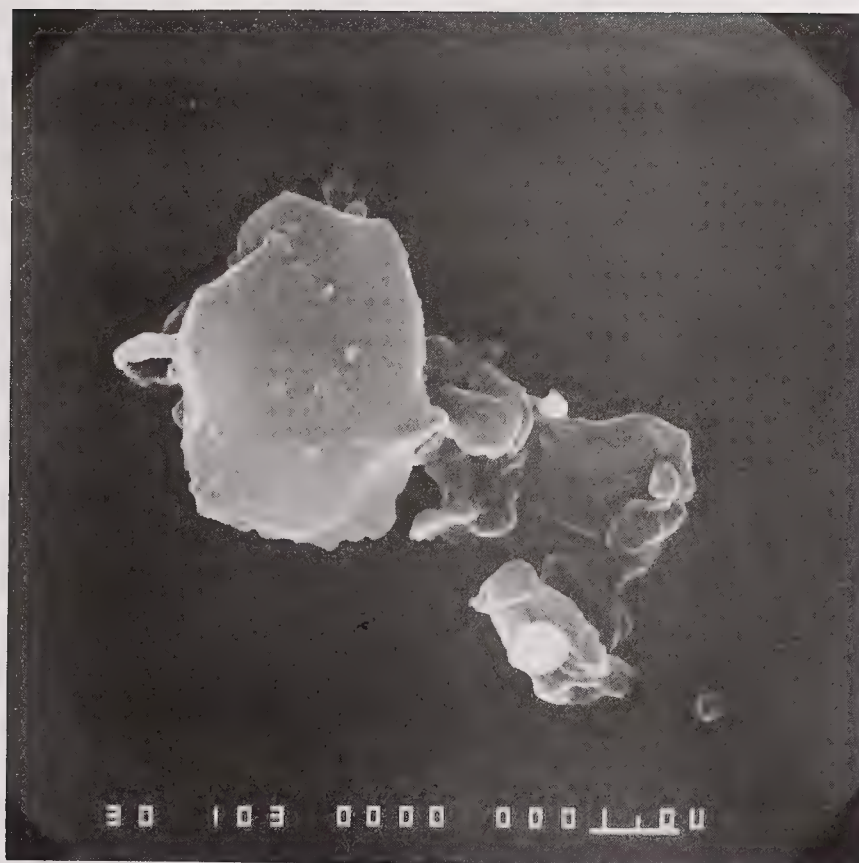


Figure 60. SEM micrograph of particle. Magnification 10,000x; scale bar in lower right of micrograph indicates 1 μm length. Sample: Si 1001. Lab No. 4.

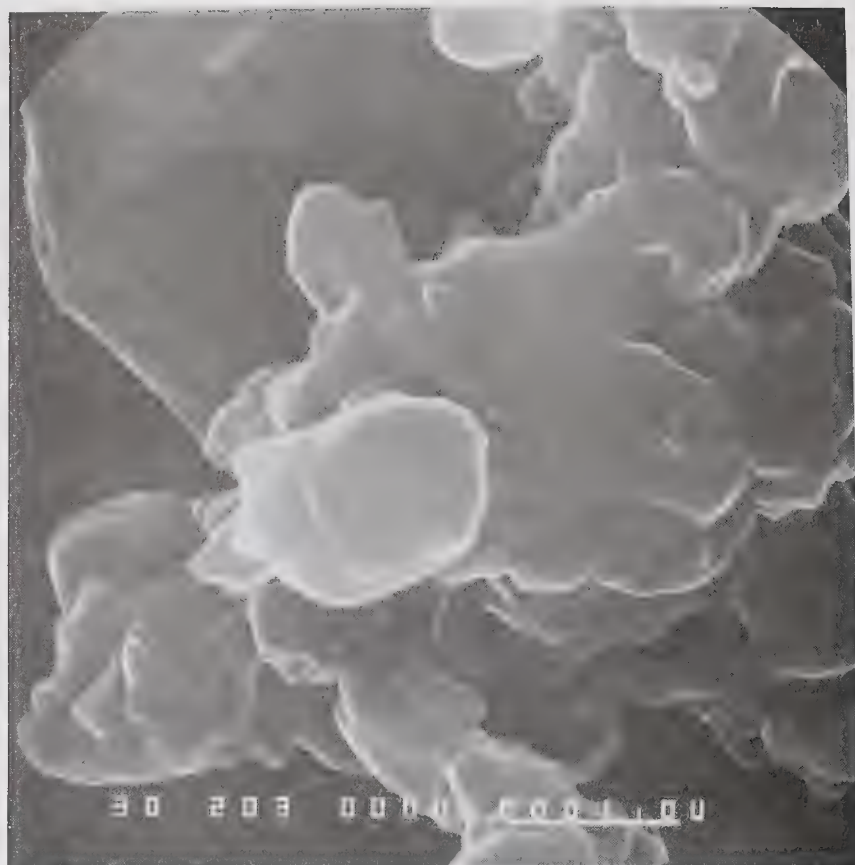


Figure 61. SEM micrograph showing detail of particle. Magnification 20,000x; scale bar in lower right of micrograph indicates 1 μm length. Sample: Si 1001. Lab No. 4.

5.2.1.5. Yttria-Zirconia Powder

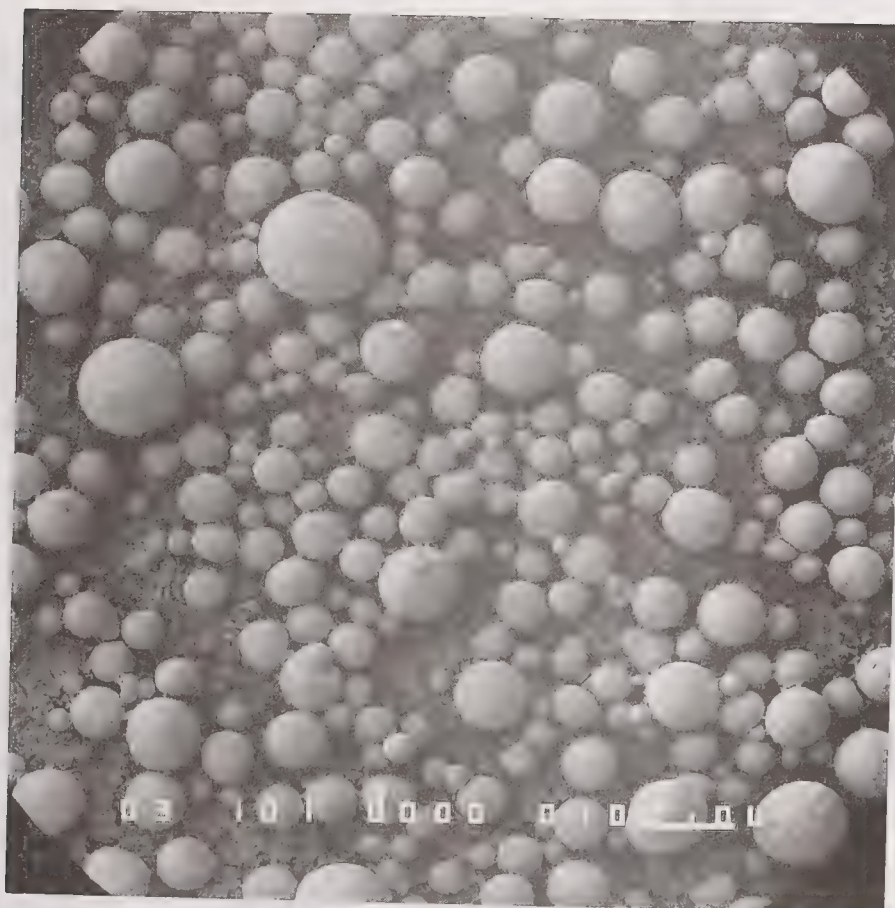


Figure 62. SEM micrograph of spray-dried powder as-received. Magnification 100x; scale bar in lower right of micrograph indicates 100 μm length. Sample: YSZ AEO 11. Lab No. 4.



Figure 63. SEM micrograph of single spray-dried particle. Magnification 1000x; scale bar in lower right of micrograph indicates 10 μm length. Sample: YSZ AEO 11. Lab No. 4.



Figure 64. SEM micrograph of powder after dispersal in water and partial disruption of agglomerated particles. Magnification 5000x; scale bar in lower right of micrograph indicates 10 μm length. Sample: YSZ ORO 12. Lab No. 18.



Figure 65. TEM micrograph of primary particles. Sample: YSZ 0514.
Lab No. 22.



Figure 66. TEM micrograph of primary particles. Sample: YSZ 0269.
Lab No. 22.

5.3. PARTICLE MORPHOLOGY - QUANTITATIVE DATA

5.3.1. Scanning Electron Microscope, Wadell Diameter

The Wadell diameter is defined as the diameter of a circle having the same projected area as the image of the particle.

Image analysis of scanning electron microscope (SEM) micrographs yields a number based size distribution, from which a mean thickness can be calculated as

$$\langle x \rangle_N = \Sigma x dN / \Sigma dN, \quad (3.3)$$

where dN is the number of particles with the interval of linear dimension x to x+dx. A mass- or volume-based distribution can be estimated by assuming that the out-of-plane dimension is equivalent to the in-plane dimension. The mean size obtained for the estimated mass distribution is

$$\langle x \rangle_M = \Sigma x^4 dN / \Sigma x^3 dN \quad (3.4)$$

where dN is the number of particles with the specific characteristic linear dimension between x and x+dx, and where sums over all sizes is implied. The geometric standard deviation is defined with respect to a lognormal distribution.

5.3.2. Silicon Nitride Reference Powder

Table 5.3.2.1 Experimental Mean, Number Distribution

Lab No.	No. of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
23	1	--	--	0.4	--

Table 5.3.2.2 Experimental Mean, Mass Distribution

Lab No.	No. of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
23	1	--	--	1.39	--

Table 5.3.2.3 Standard Deviation (σ_g) for the Size Distribution

Lab No.	No. of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
23	1	--	--	2.2	--

5.3.3. Silicon Nitride Test Powder

Table 5.3.3.1 Experimental Mean, Number Distribution

Lab No.	No. of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
23	2	0.4	0.5	0.45	0.05

Table 5.3.3.2 Experimental Mean, Mass Distribution

Lab No.	No. of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
23	2	1.03	1.06	1.05	0.02

Table 5.3.3.3 Standard Deviation (σ_g) for the Size Distribution

Lab No.	No. of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
23	2	1.8	2.0	1.9	0.1

5.3.4. Silicon Carbide Powder

Table 5.3.4.1 Experimental Mean, Number Distribution

Lab No.	No. of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
23	3	0.32	0.41	0.36	0.05

Table 5.3.4.2 Standard Deviation (σ_g) for the Size Distribution (Number)

Lab No.	No. of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
23	3	1.64	1.96	1.79	0.16

Table 5.3.4.3 Experimental Mean, Mass Distribution

Lab No.	No. of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
23	3	0.79	0.90	0.84	0.06

Table 5.3.4.4 Standard Deviation (σ_g) for the Size Distribution (Mass)

Lab No.	No. of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
23	3	1.61	1.75	1.68	0.070

5.3.5. Silicon Powder

Table 5.3.5.1 Experimental Mean, Number Distribution

Lab No.	No. of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
23	6	1.06	1.23	1.15	0.06

Table 5.3.5.2 Standard Deviation (σ_g) for the Size Distribution (Number)

Lab No.	No. of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
23	6	2.10	2.33	2.24	0.09

Table 5.3.5.3 Experimental Mean, Mass Distribution

Lab No.	No. of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
23	6	4.89	7.15	6.14	0.83

Table 5.3.5.4 Standard Deviation (σ_g) for the Size Distribution (Mass)

Lab No.	No. of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
23	6	1.78	2.04	1.94	0.09

5.3.6. Yttria-Zirconia Powder

Table 5.3.6.1 Experimental Mean, Number Distribution

Lab No.	No. of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
23	1	--	--	0.3	--

Table 5.3.6.2 Experimental Mean, Mass Distribution

Lab No.	No. of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
23	1	--	--	2.27	--

Table 5.3.6.3 Standard Deviation (σ_g) for the Size Distribution

Lab No.	No. of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
23	1	--	--	2.88	--

5.3.7. SEM, Size Measured as Maximum Particle Dimension

5.3.7.1. Silicon Nitride Reference Powder

Table 5.3.7.1 Experimental Mean, Number Distribution

Lab No.	No. of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
13	12	0.6	1.0	0.8	0.1

Table 5.3.7.1.1 Experimental Mean, Mass Distribution

Lab No.	No. of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
12	1	--	--	1.5	--

5.3.7.2 Silicon Nitride Test Powder

Table 5.3.7.3 Experimental Mean, Mass Distribution

Lab No.	No. of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
12	1	--	--	1.2	--

5.3.7.3. Silicon Powder

Table 5.3.7.3 Experimental Mean, Mass Distribution

Lab No.	No. of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
12	1	--	--	2.1	--

5.3.7.4. Yttria-Zirconia PowderTable 5.3.7.4 Experimental Mean, Number Distribution,
As-Received Powder

Lab No.	No. of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
18	6	27.49	39.36	33.98	4.14

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6. CHEMICAL COMPOSITION OF POWDERS

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6.1 INTRODUCTION

The importance of measurement and control of the chemical composition of starting powders for advanced structural ceramics is widely recognized, but accurate determination of important chemical species remains problematical. Two examples are:

- (1) the presence of oxide in SiC and Si₃N₄ powders which alter their sinterability, and
- (2) the presence of Ca, and other glass formers, in Si₃N₄ which reduce the high-temperature strength of the ceramic.

Chemical analyses which require the dissolution of the material are complicated by the general chemical refractiveness of the compounds so that failure to recover all of the component of interest as a reaction product and contamination by the apparatus, such as pressure bombs, are problems which require special attention.

The IEA/Annex II Subtask 2 study provides a comparison of methods used for the determination of major constituents, major impurities (> 100 ppm), trace elements, and volatile adsorbed species, such as water. Results of determination of volatilized absorbed species are presented in Chapters 11 and 12. The present discussion provides a summary description of the analytical methods, compares results within a method and between methods for a given component, and examines the possible sources of variance in the analytical procedures used to determine nonvolatile components of the powders.

Analytical Procedures

Analytical procedures generally consist of five discrete steps:

1. Sampling
2. Sample preparation
3. Measurement
4. Data processing
5. Analytical process correction, involves testing, controlling and eventually correcting one or more of the above processing steps.

Sampling and Sample Preparation

A description of the preparation of the IEA samples (vials) is given in Section 1.3. of this report. The extraction of analytical samples from the IEA samples was carried out at the discretion and according to the method of the analyst. Use of the entire IEA sample was recommended whenever possible, otherwise random sampling from the IEA sample was recommended.

Sampling errors, such as contamination and fractionation, may arise at three steps in sampling process: (1) during the manufacture and handling of the powder, (2) during the taking or the division of samples for testing, and (3) during the taking of analytical samples by the analyst. Errors associated with "(1)" are expected to appear in overall large variability in the results for the population of all laboratories reporting results; whereas "(2)" and "(3)" are expected to appear as a large excursion of the result for a single group of samples or of a single sample, respectively, from the mean value for all samples. Since a record of the powder division history was maintained during the preparation of IEA samples, the validity of "(2)" may be inferred if two or more samples from the same division pathway were analyzed in the round-robin. A type "(3)" error may be expected if types "(1)" and "(2)" can be ruled out, or if the laboratory in question has run replicate samples from the same IEA sample and has obtained widely different results. A type "(3)" error is likely to result if the analyst did not carefully sample randomly throughout the IEA sample but instead extracted a grab sample. However, a type "(3)" error can be easily confused with an error arising in Step 2, Sample Preparation.

Measurement, Data Processing and Testing

The variance associated with Step 3, Measurement, can be estimated from replicate runs on the same sample. Such errors can be reduced in laboratory practice by frequent running of a control sample and the use of control charts to verify instrument and operator performance. Similarly, with regard to Step 4, Data processing, replicate readings of a chart, meter or digital display of an instrument output provide an estimate of the variance of the reading of the instrument response to the phenomenon measured.

An error occurring in Step 5, Analytical Process Correction, most likely appears as a bias which is evident in the deviation of all results of a particular laboratory. However, since analytical standards are not presently available for the materials used in this study, assessment of this bias is not generally possible. Further, standard analytical practices have not been established in many cases, nor are common; nor the best practices widely known or used. Consequently, we were too frequently informed that analysts used the same practice for all materials, with little regard to chemical and analytical problems introduced. It is hoped that the present study, and the comparison of practices which it has made possible, will result in an improvement in analytical practices for Si_3N_4 , SiC , Si and ZrO_2 .

Methods for Chemical Characterization

The methods presented for each type of analysis are arranged in the general order: whole sample methods, such as neutron activation analysis and x-ray fluorescence; spectrochemical methods requiring dissolution of the sample, with absorption followed by various emission types; electrochemical methods; ion chromatography; gravimetry; and volumetric analysis.

The various spectrochemical methods can be classified on the basis of (1) whether the mode of analysis uses the characteristic emissions or absorptions by the sample, (2) type of excitation, (3) form of the sample preparation, and

(4) the analytical range of the analysis, including the number of elements and the concentration range. The methods included in this study are compared in Table 6.1.1 with respect to the four categories. The section in which the method is discussed in this report is given in the left hand most column along with the acronym. The acronyms ICP (inductively coupled plasma) and DCP (dc plasma jet) when used alone are assumed to refer here to their use in the emission mode; when used in the absorption mode, these acronyms are usually prefixed to AAS (atomic absorption spectroscopy). Flame-AAS is used to designate flame absorption spectroscopy.

Table 6.1.1 Comparison of Spectrochemical Methods for Elemental Analysis

METHOD	TYPE	EXCITATION	SAMPLE PREP'N	RANGE
XRF (4.2.1.1.)	E	X-rays	Homogeneous powder compacts,	Major components Major impurities Z > 22 (Ti) air, 13 (Al) in He, 9 (F) in vac.
OES (4.1.1.3.)	E	Electric arc	Powder sample	General, use; most elements; sensitivity varies
AAS (4.2.1.2.)	A	Flame DCP jet	Solution	General use; most elements; sensitivity varies
ICP (4.2.1.3.)	E or A	Inductively coupled plasma	Solution	General use; most elements; sensitivity varies
DCP (4.2.1.4.)	E or A	DC plasma	Solution	General use; most elements; sensitivity varies

E - emission, A - Arc, Z - atomic number

Some methods described here have a sufficiently large dynamic range to enable them to be used for more than one range of concentrations. These instruments are discussed once for one component range and mentioned with supplemental descriptions as needed for other ranges for which they are relevant. For each component range, the instruments used in the study are described and then the measurement results are presented and compared.

7. MAJOR CHEMICAL COMPONENTS

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7.1. METHODS FOR SILICON IN SILICON NITRIDE, SILICON CARBIDE AND SILICON

The principal methods investigated were inductively coupled plasma-emission spectroscopy, dc plasma-emission spectroscopy and gravimetric analysis. Results also were reported for measurements by x-ray fluorescence spectrometry, optical emission spectroscopy, atomic absorption spectrometry and coulometric titration.

7.1.1. X-Ray Fluorescence Spectrometry (XRF)^{3,4}

Use and Significance

X-ray fluorescence spectroscopy is primarily used for semiquantitative and quantitative determination of major elemental constituents and major impurities with atomic number greater than 9 (vacuum operation). XRF is capable of performing rapid, high-quality multielement determinations on a single sample preparation. Reference standards are required for the conversion of x-ray intensities to concentrations.

Instrument and Experimental Parameters

The XRF method uses x-ray to excite the characteristic x-ray fluorescence of elements in a sample. Two methods are used to analyze the fluorescent radiation:

- (1) In energy-dispersive or fluorescence analysis, the spectrum of fluorescent radiation emitted by the sample is separated on the basis of energy using a Si(Li) detector and a multichannel analyzer (MCA). This method is widely used in industry.
- (2) In wavelength-dispersive analysis, the fluorescent radiation from the sample is collimated and directed on to an analyzing crystal. Radiation diffracted by the crystal is detected by a counter which is moved along the spectrometer circle.

The excitation radiation must be of slightly higher frequency (shorter wavelength) than that of the characteristic fluorescence of interest. In practice, the excitation radiation is produced using an x-ray tube with a tungsten or other heavy metal target for heavy elements or with a chromium target for light elements. Energy-dispersive spectrometers may use the monochromatic fluorescent radiation from a secondary fluoresces rather than the direct radiation from an x-ray tube.

The dynamic range of XRF is 0.2 Å (62 keV) to 20 Å (0.6 keV). Resolution of an energy-dispersive spectrometer improves with shorter wavelength or greater energy. For wavelength-dispersive spectrometers the reverse is true.

Preparative methods reported are summarized in Table 7.1.1.1. Although XRF can be carried out nondestructively, the most accurate results are obtained by dissolving the sample in a glass matrix. Typically the sample is mixed with a material such as lithium tetraborate, with the possible addition of a heavy absorber such as barium carbonate, fused and cast into a glass bead. Pressed powder samples may also be used, but the uniformity of the mixing in this case may be somewhat less precise. The principle in sample preparation is to obtain samples which will totally absorb all of the incident x-rays and which will contain a uniform dispersion of the fluorescing elements.

Table 7.1.1.1 Preparative Methods Used for Total Silicon Analysis by X-ray Fluorescence Spectrometry

LAB	METHOD
5	First melt with $\text{Li}_2\text{CO}_3 + \text{B}_2\text{O}_3$; second melt after addition of more B_2O_3 and of LiI .
25	Oxidation with CaO ; $\text{Li}_2\text{B}_4\text{O}_7$ bead.

Calibration and Standards

To accurately derive elemental concentrations from the measured fluorescent x-ray intensities, it is necessary to correct the intensities for matrix absorption and enhanced excitation. The first effect arises from secondary absorption of the fluorescent radiation by the sample. The second effect results from the stimulated emission of secondary fluorescent radiation by the primary fluorescent emission of the sample. Corrections can be made for these effects by use of an empirical calibration curve or by fundamental calculation of parameters.

A variety of rocks, minerals and ores are available from the National Institute of Standards and Technology as compositional references for both multi-component and impurity concentrations. In addition, SRM 477 is a glass fluorescence source which is used for determining detector window absorption in semiconductor x-ray spectrometers.

7.1.2 Atomic Absorption Spectrometry (AAS)

Use and Significance

Flame atomization and electrically heated furnace atomization AAS have become among the most widely used analytical procedures for the determination of metallic elements (at least 67 elements). Inductively coupled plasma and dc plasma jets, see Sections 7.1.3. and 7.1.4. below, respectively, can also be used as atomization sources for this method. Flame atomic emission spectroscopy also exists, but since it was not used in this study, it will not be considered further here.

Instrumental and Experimental Parameters^{1,2}

Chopped monochromatic light is passed through the flame to a detector. The source radiation consists of a line spectrum of the element of interest. The sources commonly used for AAS are (1) hollow-cathode discharge tubes, (2) metal-vapor arc lamps, and (3) electrodeless discharge tubes. A linear flame with multiple beam passes or more than one linear flame may be used to increase the pathlength through the flame. Most flame AAS studies have been performed with air-acetylene flames, although oxyhydrogen, oxyacetylene, nitrous oxide-acetylene and other flames have been used. Continuum sources also can be used where high sensitivity is not required and if the monochromator has a narrow spectral bandwidth. In analytical work, the fraction of radiation absorbed at the line center is measured rather than the integrated intensity. Thus, a narrow line source is desirable. Single beam optics have been used on most commercial systems. This requires a well-regulated source of power supply and a longer warm up time for the source lamp. Data output is usually to a meter, recorder, digital readout, or computer.

Parameters which may influence performance include the following: aspirating gas flow rate; fuel-to-oxidant flow ratio; solution flow rate into the flame; orifice size; temperature of chamber, if a constant temperature chamber is used; height of measurement in the flame; entrance optics, only if optics can be varied; monochromator slit width; light power source; and pathlength through flame.

The material to be analyzed is dissolved and nebulized into a gas stream which carries the aerosol into the flame. Since ceramic powders are often not easily decomposed, drastic decomposition conditions such as fluxes or strong acids must be employed. In geochemical practice, it is customary to decompose the material with a borate melt, followed by dissolving in a solution buffered with citric or tartaric acid. These solutions have been reported to be unstable.³ One laboratory (14) which reported the use of AAS decomposed silicon nitride samples in NaOH-Na₂O₂ melts. Lab (13) reported that strong NaOH solutions have been presumed to yield unstable AAS measurements and to attack the burner.

Running a blank solution to establish a correction for background absorption is recommended if a solution has a high ionic strength. A background correction can also be made by measuring the intensity of a second nonabsorbed line, such as a signal from a deuterium lamp.

Results and Discussion

The average result for AAS is compared with other spectrochemical methods in Table 7.1.2.1. One laboratory reported results for two samples of the SNR Si₃N₄ and one sample of SNT Si₃N₄ in this study. Good agreement between results was found with respect to the samples for the SNR powder and with respect to results reported for other methods. Although the high precision for the analysis of N in the SNR powder by AAS and the close agreement with the XRF determination lend encouragement toward the further use of AAS, the limited number of determinations reduces the overall confidence in the present result.

Conclusions

Determinations of Si content of Si_3N_4 by AAS are comparable to those obtained with XRF, ICP and DCP. However, the number of results was insufficient for an adequate evaluation of this method.

TABLE 7.1.2.1 Determination of Si in Silicon Nitride and Silicon Carbide by Spectrochemical Methods. Concentrations are in percent by weight.

MATERIAL	METHOD	NUMBER OF		MEAN	STANDARD DEVIATION
		LABS	SAMPLES		
Si_3N_4 (SNR)	XRF	2	2	59.7	
	AAS/Flux	1	2	59.5	0.0 ^a
	ICP/Flux	2	5	59.2	0.8
	ICP/Acid	1	1	59.4	
	ICP/NS ^b	1	2	58.7	1.0
	DCP/Flux	2	4	58.8	1.7
Si_3N_4 (SNT)	XRF	2	3	59.5	0.3
	AAS/Flux	1	1	60.0	
	ICP/Flux	2	5	59.8	0.5
	ICP/Acid	1	1	60.2	
	DCP/Flux	2	2	61.0	1.9
SiC	XRF	1	6	69.1	0.2
	ICP/Flux	2	4	69.2	0.2
	ICP/Acid	1	1	70.1	

^a Could not be estimated

^b NS- not specified

7.1.3. Inductively Coupled Plasma (ICP)

Use and Significance

Inductively coupled plasma-atomic emission spectroscopy (ICP) uses the excitations of atoms in a plasma to obtain their characteristic emissions. It has found wide industrial application for the quantitative elemental analysis of materials since it provides a determination of major, minor and various trace elements. The material to be analyzed is dissolved, and the solution is then injected as an aerosol into the axial channel of a toroidal argon plasma. Dissolution of the sample and preparation of solutions are critical steps in the accurate use of this method.

Instrumental and Experimental Parameters^{4,5}

The operating parameters are rf power, gas flow rates to the nebulizer and the plasma. Ambient conditions in the axial channel may be characterized by gas temperatures of 4000-6000 K, ionization temperatures of approximately 8000 K, and electron number densities on the order of 10^{15} cm^{-3} . Under these conditions a high yield of singly charged, monatomic ions is obtained for most elements. Quantitative determination of an element requires the use of spectral line intensities which are selected by optimizing the criteria of low detection limit, low background equivalent concentration, and absence of interfering lines of major elements.

Sample preparation is perhaps the most important experimental consideration since accurate determinations require the quantitative recovery of the elements of interest from the solid samples. Analysis of ceramic materials which are chemically very stable may pose special analytical problems and most of the analytical error may be assignable to the preparation of analytical samples. Due to the drastic conditions required to achieve dissolution, the determination of blank values is important.

Two approaches for dissolution of ceramic samples were used in this study:

1. fusion of the sample in a borate or carbonate, followed by the dissolution of the fluxed material in an acid solution;
2. dissolution of the sample in an acid mixture, such as HF plus HNO_3 .

Fluxes used for the analysis of Si-based materials included sodium carbonate with boric acid and lithium metaborate (LiBO_2).

Acid dissolution was carried out in a pressure bomb; both teflon or glassy carbon were investigated as liners for the bombs. The various methods are summarized in Table 7.1.3.1.

Results and Discussion

For each material analyzed in this study, three or more labs reported determinations, two of which used flux dissolution of the materials. Good

agreement between results was found in all cases, as evidenced by the results given in Table 7.1.2.1.

Table 7.1.3.1 Preparative Methods for Analysis of Total Silicon in Silicon Nitride, Silicon Carbide and Silicon by Inductively Coupled Plasma Emission Spectroscopy

LAB #	METHOD
1	Not specified.
6	Lithium metaborate (LiBO_2) fusion at 1000 °C for 2h; quenched into a solution of H_2O_2 + HNO_3 + tartaric acid.
15	HF + HNO_3 , 150 °C, pressure decomposition.
21	Decomposition with Na_2CO_3 + H_3BO_3 .

Conclusions

Determinations of silicon content of Si_3N_4 and SiC by ICP are comparable to those obtained with XRF, AAS and DCP. Flux and acid decomposition methods also appeared to yield comparable Si concentrations in the cases of the two Si_3N_4 analyses and of the one SiC . However, the number of results reported was insufficient for an accurate comparison of methods.

7.1.4. Direct-Current Plasma (DCP) - Emission Spectroscopy

Use and Significance

Direct-Current Plasma Jet-Emission Spectroscopy (DCP) is very similar to the flame emission spectroscopy with the exception that the excitation source is a dc plasma jet. An analyte solution is nebulized, similar to the manner of ICP, or a finely ground powder is dispersed in a gas stream and carried into the plasma jet. Chambers yielding very stable arcs and good gas mixing have been built in recent years. As with ICP, dissolution of the sample and preparation of solutions for analysis are critical steps in this method.

Instrument and Experimental Parameters⁶

The plasma jet is formed between a graphite ring anode and a graphite ring cathode with the jet passing through the orifice of the cathode. Nitrogen, air or inert gases may be used for the plasma. The sample is injected along the axis of the jet. Stabilizing gases are introduced into the water-cooled chamber which encloses the space between the anode and cathode. Special burner designs have been developed to achieve a stable plasma, to evaporate an aerosol completely, and to achieve sufficient penetration of the aerosol into the plasma by turbulent mixing of the aerosol into the gas stream.

The emission spectra are analyzed with a prism or grating monochromator. Photodetection is usually carried out with a vacuum single-stage or, more commonly, multistage phototube. The output current is amplified and then fed to a readout device.

Instrumental parameters include flow-rates for the nebulizing gas, carrier gas, plasma gas and stabilizing gas; voltage and current to the plasma jet; as well as parameters of photodetector and readout device.

Experimental considerations are similar to those for ICP. In this study, only flux decomposition was used to prepare samples for DCP analysis. Experimental conditions for dissolution and analysis of Si_3N_4 were discussed by Davis and Merkle⁷ and by Czupryna and Natansohn⁸.

Results and Discussion

DCP was used for analysis of total Si in Si_3N_4 in this study. Examination of the results given in Table 7.1.2.1 indicates that measurements by DCP are in agreement with those obtained by XRF and ICP; however, the precision of the result by DCP is perhaps worse than that for the ICP results: DCP gave a relative standard deviation of 3%, whereas, that by ICP was about 1%.

Conclusions

Determinations of silicon content of Si_3N_4 by DCP are comparable to those obtained with XRF and ICP. The between laboratory precision obtained with this method appears to be somewhat poorer than that for XRF and ICP. However, the data are inadequate in all cases for an accurate comparison of precision to be made.

7.1.5. Gravimetry

Use and Significance

Gravimetry refers to the determination of a component by isolation of it in a solid form which can be weighed, thus providing an independent determination of that component. Estimation of the amount of a component by subtraction of the amounts of all other components from the total weight of the sample does not constitute an independent measurement, albeit such an estimate may be useful for the purpose of comparison.

Components, such as Si, which can be readily converted to a stable oxide that can be recovered and weighed, may be determined by gravimetry. Much depends upon the procedure required to separate the component and to precipitate or collect in such a manner as to exclude other chemical species.

Instrument and Experimental Parameters

No special instrumental considerations are required.

Decomposition of the nitrides and carbide was carried out in borate or

carbonate fluxes, followed by dissolution in nitric or hydrochloric acids and precipitation of SiO_2 or fumed in perchloric acid to form SiO_2 .

Results and Discussion

Results are generally consistent with those from spectrochemical methods. The large standard deviation for Si in SiC suggests that all of the Si may not have been effectively recovered with the chemical methods used. Refer to Table 7.1.5.1.

Conclusions

Data were insufficient for statistical analysis of variances in the cases of SNT Si_3N_4 and SiC. For the two silicon nitride powders gravimetry yielded results which were consistent with those from the spectrochemical methods. The gravimetric result for SiC was about 2% low with respect to those results and has much lower precision. The reliability of this method for SiC needs to be established.

TABLE 7.1.5.1 Determination of Si in Silicon Nitride and Silicon Carbide by Gravimetric Determination as SiO_2 . Concentrations are in percent by weight.

MATERIAL	LABS	NUMBER OF		STANDARD DEVIATION
		SAMPLES	MEAN	
Si_3N_4 (SNR)	3	15	59.42	0.50
(SNT)	2	3	59.32	0.19
SiC	2	3	67.66	1.56

7.2 METHODS FOR NITROGEN IN SILICON NITRIDE

Nitrogen in silicon nitride was determined primarily by combustion analysis. Some determinations were made using electrochemical means of detection and by Kjeldahl analysis, but these also required decomposition of the powder and, hence, were very similar to the combustion method in regard to decomposition chemistry.

7.2.1. Combustion Method

Use and Significance

Combustion, or fusion, is used when the chemical component can be liberated in a gaseous form from the sample by a decomposition reaction. Usually, a reactive flux is used. The quantity of gaseous product obtained is determined from a property of the gas. This method is widely used in industrial

laboratories for the analysis of C, O, N and S in Si_3N_4 , SiC and Si.

Instrument and Experimental Parameters

Measurement of quantity of gas liberated was not clearly specified in most cases but is presumed to have been by the usual method of measuring the thermal conductivity of the product gas. Manufacturers of instrumentation for this method supply N-containing pellets for calibration of their instruments.

Method of decomposition was not identified by most labs. Two labs reported use of graphite crucibles, with one of those labs reporting the encapsulation of the sample in Sn/Ni and comparison against an Si_3N_4 laboratory standard.

Results and Discussion

The average results for determinations of N in SNR Si_3N_4 , SNT Si_3N_4 , SiC and Si by combustion analysis are compared in Table 7.2.1.1 with results obtained by other methods.

For the case of SNR, 4 labs reported a total of 21 determinations which were in very close agreement (s.d. of 0.24). Two other labs reported results which were not included here. One of the laboratories reported a single low value of 32.10, which is suspected to be due to a very large measurement error from loss or failure to recover all of the N present. The other laboratory reported results for 11 samples, a single determination for each sample, with a mean of 40.80 and s.d. of 0.48. From the standpoint of stoichiometry, a value greater than 39.94 is not expected. Further, the difference between the two mean was significantly different. Thus, the high results are not included in Table 7.2.1.1.

TABLE 7.2.1.1 Determination of N in Silicon Nitride, Silicon Carbide, Silicon and Yttria-Zirconia by Combustion. Concentrations are in percent by weight.

MATERIAL	DETECTION	LABS	NUMBER OF		STANDARD DEVIATION
			SAMPLES	MEAN	
SNR	TC	9	15	38.23	0.29
SNT	TC	8	19	38.95	0.78
SiC	TC	4	6	0.076	0.007
Si	TC	4	7	0.04	0.03
YSZ	TC	1	2	0.027	0.004

Conclusions

Determination of the N content of silicon nitride materials by decomposition followed by detection and measurement by thermal conductivity measurements is capable of high precision. However, care must be taken to recover all of the N and to determine that the instrument is correctly calibrated. Development of a reference material for verification of measurements is desirable.

7.2.2. Kjeldahl Analysis

Use and Significance

Kjeldahl analysis is a neutralization titration method which is the standard means for the determination of N in a variety of materials. The neutralization technique is carried out using a standard solution of strong acid or strong base.

Instrument and Experimental Parameters

Nitrogen in the material is liberated as ammonia, which is recovered and the amount determined. Recovery and determination are accomplished either by:

- (1) collecting the ammonia in a known quantity of standard acid and back-titrating with a standard base; or
- (2) collecting the ammonia in an unmeasured excess of boric acid and the borate ion formed is titrated back to boric acid with a standard solution of hydrochloric acid.

Decomposition of the nitrogenous material in hot sulphuric acid is used in many cases. However, in this study, one laboratory reported the use of a LiOH flux and two other laboratories reported the use of hydrofluoric acid or hydrofluoric acid with nitric acid in a pressure bomb.

Results and Discussion

For the SNR, a total of two determinations were reported by two laboratories, with a mean value of 38.66. However, for the SNT, 11 determinations were reported by three laboratories which used four samples. The mean value and standard deviation were 38.91 and 0.12, respectively.

These results are comparable to those obtained with the other methods of detection which are preceded by sample decomposition since similar decomposition chemistries were used. The precision of this method appears to be slightly better than that obtained by decomposition followed by measurement of the N₂ with thermal conductivity.

Conclusions

Nitrogen concentrations were determined with good precision in silicon nitride. The somewhat more extensive chemical operation required by Kjeldahl analysis as compared to the decomposition-gas detection methods may be more

than offset by the better precision obtained. This method may be preferred for high-quality analyses or when reference measurements are required. Further, testing of the method is recommended to establish a fully evaluated procedure and to obtain good estimates of the sources of variance.

7.2.3. Electrochemical Methods

Under the general category of Electrochemical Methods are included coulometry, specific ion potentiometry, and potentiometric and argentometric titrations.

Use and Significance

Electrochemical methods do not find as wide application as the spectrochemical methods described above, since after desolution of the material, further chemical steps may be required to remove interfering chemical species. However, where an electrochemical method has been established in a laboratory, its use may yield very reliable results. Furthermore, the determination may be directly related to the concentration of the analyte present, without the introduction of a calibration.

Instrument and Experimental Parameters

Coulometry

Coulometry or coulometric titrations measure the quantity of electricity (in Coulombs) passed to an electrode by a chemical reaction involving the ion of interest. Two techniques are in use, 1. controlled potential coulometry, 2. constant current coulometry.

If the reaction is 100% current efficient, then the passage of one Faraday of electricity will cause the reaction of one equivalent of material. The concentration of an ion is usually determined by one of the following methods:

- a. measurement of the current integrated over the elapsed time,
- b. measurement of the change in weight of an electrode due to plating out of an element,
- c. measurement of the charge transfer in the opposite direction in a subsequent circuit after the coulometric reaction of interest has been carried out.

The method is particularly suited and accurate in the range from milligram to microgram quantities of material, hence it is suitable for trace element analysis. It is highly useful for the determination of volatile reactants (Cl, Br and I) and unstable reactants (Ti^{5+} , Cr^{2+} , Cu^+ and Ag^{2+}).

Standards are not generally required since the determination is referable to fundamental quantities (current and time).

Ion Selective Potentiometry

Ion selective potentiometry measures the activity of a specific ion - i.e., the thermodynamically effective free-ion concentration and not the total

concentration - by direct measurement of an electrode potential. In this study, specific ion potentiometry was used for the determination of F^- in silicon nitride.

Electrodes for specific ionic activity determinations can be grouped as:

- a. glass electrodes,
- b. solid-state and precipitate electrodes,
- c. liquid-liquid and membrane electrodes,
- d. enzyme and gas sensing electrodes.

All of these yield potentials that can be adequately described by the classical Nernst equation or its modifications.

Selective ion electrodes are subject to interferences due to method and electrodes. Method interferences occur when the sample prevents the probe from sensing the ion of interest. For example, in acid solutions, F^- forms complexes with H_3O^+ which are not measured by the electrode. Electrode interferences arise when the electrode responds to ions in the solution other than the ion of interest.

Specific ion activity is obtained by use of a calibration chart to convert measurements of pH or mV or by direct reading from a calibrated meter. For concentrations less than 10^{-4} M, calibration procedures must use solutions of known activity or concentration, depending on which parameter is required.

Potentiometric Titration

Changes in electromotive force (emf) of a cell with the addition of a titrant can be used to locate the equivalence point of a reaction. Potentiometric titrations are particularly useful when detection of the endpoint of the titration is obscured by turbidity, fluorescence, opaqueness, highly colored solutions or when a visual indicator is not available. Automatic titrators are available which permit precise control and continuous recording of the process to be carried out.

The solution containing the unknown is titrated against a standard solution until the equivalence point, as determined by a rapid change in electrode potential, is reached. The concentration of the unknown is calculated from the volume of standard solution added. The determination is expedited, if the approximate concentration of the unknown is known, by adding standard solution rapidly until in the neighborhood of the equivalence point and then carefully titrating through the equivalence point. Automated systems can be programmed to carry out such a procedure.

Argentometric Titration

Argentometric titrations are a specific category of potentiometric titration in which silver-related equilibria are employed. Two specific equilibria, are associated with the solubility of $AgCl$ and with the formation of the $Ag(CN)_2^-$ complex. In the case of $AgCl$ equilibrium the emf associated with the Cl^- concentration is measured with an electrode "of the second kind", i.e., an

electrode in which a cation is in equilibrium with its sparingly soluble salt. At the equivalence point of the titration:

$$[\text{Ag}^+] = [\text{Cl}^-] = \sqrt{K_{\text{sp}}}$$

Ion selective electrodes can be used in conjunction with the titration process to determine the cation (Ag^+) or anion concentrations.

Experimental considerations are similar to those for potentiometric titration.

Results and Discussion

One laboratory reported the use of potentiometric titration to determine the concentration of nitrogen in both types of silicon nitride. The samples were reacted with a LiOH flux and the nitrogen recovered as ammonia. In case of SNR, three samples were analyzed; two samples were found to have N-concentrations comparable with values found by other methods, mean of 37.9, but one sample was significantly low. For SNT, two specimens of the one IEA sample were analyzed with a mean of 38.15.

Conclusions

The number of results are insufficient to permit valid comparisons and inferences to be made. Further evaluation of this method is needed.

7.3. METHODS FOR TOTAL CARBON IN SILICON CARBIDE

Total carbon in silicon carbide was carried out primarily by combustion analysis. Coulometric titration also was used. One gravimetric measurement was reported.

7.3.1. Combustion (See 7.2.1.)

Instrument and Experimental Parameters

Carbon in the sample which is liberated as CO_2 is determined by measuring the infrared absorption of a precise IR wavelength. A reaction is used which liberates C as CO or CO_2 . The CO is further oxidized to CO_2 before reaching the IR detector.

Results and Discussion

The average result for determination of total carbon in SiC is presented in Table 7.3.1.1, along with determination of carbon impurities in silicon nitride, silicon and yttria-zirconia. The limited amount of data does not permit an accurate estimate of the sources of variance - within laboratory (sample, determination) and between laboratories. Two laboratories reported multi-determinations on a single IEA sample, with a standard deviation of about 0.05. The laboratory reported single measurements on two samples with a standard deviation of about 0.4. Approximately, half of the observed variance

may be assigned to between-laboratory variance. The approximately equally large sample-to-sample and between-laboratory variances suggest that either significant sample-to-sample inhomogeneity was present or that

TABLE 7.3.1.1 Determination of C in Silicon Nitride, Silicon Carbide, Silicon and Yttria-Zirconia by Combustion. Concentrations are in percent by weight.

MATERIAL	DETECTION	LABS	NUMBER OF SAMPLES	MEAN	STANDARD DEVIATION
SNR	IR	13	24	0.15	0.02
SNT	IR	12	23	0.13	0.03
SiC	IR	5	6	29.62	1.00
Si	IR	6	9	0.08	0.02
YSZ	IR	3	4	0.036	0.003

the significant differences existed in the effectiveness and uniformity of the chemical procedures used.

The results obtained by combustion are slightly below the C values obtained by ICP and DCP. Also, the values obtained by these two spectrochemical methods show greater sample-to-sample, between laboratory and between method consistency.

Conclusions

The present determination of total carbon in SiC by combustion is suspected of yielding results which are about 2% below the true value and shows poorer reproducibility than for its other applications in this study. Development of a chemical method yielding 100% recovery of the carbon in SiC as CO₂, standardization of the procedure and development of a certified material are recommended.

7.3.2. Electrochemical Methods - Coulometry (See 7.2.3.)

Results and Discussion

Three laboratories reported a total of 10 determinations for three samples. The carbon concentration is 30.04% by weight, with a standard deviation of 0.05. Estimate of the standard deviation for a single sample is 0.04.

Conclusions

The results obtained indicate that this method can yield highly reproducible results. Carbon values determined by coulometry for the IEA SiC samples are 30.04 ± 0.15 (ninety-five percent confidence limits).

7.4. METHODS FOR ZIRCONIUM IN YTTRIA-ZIRCONIA

Three laboratories reported results of measurements by ICP, while one reported six determinations by XRF and one reported three determinations by AAS.

Data are summarized in Section 7.6.4.

7.4.1. X-ray Fluorescence Spectrometry (See 7.1.1.)

Instrument and Experimental Parameters

Samples were prepared by fusion in lithium tetraborate.

Conclusion

Data were insufficient to establish between-laboratory variance. Further testing of this method is needed.

7.4.2. Atomic Absorption Spectrometry (See 7.2.1.)

Results and Discussion

One laboratory reported single results for three samples: mean 67.1, and standard deviation 0.1.

The values obtained by AAS are significantly less than those obtained by XRF. Possible incomplete recovery of Zr in dissolution of yttria-zirconia during preparation of analyte solutions may be a possible source of this bias.

TABLE 7.4.1.1 Determination of Major Elements in Yttria-Zirconia (YSZ) by X-ray Fluorescence. Concentrations are in percent by weight.

ELEMENT	NUMBER OF		MEAN	STANDARD DEVIATION
	LAB	SAMPLES		
Zr	1	6	68.50	0.04
Y	2	9	4.29	0.17

Conclusions

Data were insufficient for an adequate analysis of this method. However, results appear to be slightly low with respect to XRF.

7.4.3. Inductively Coupled Plasma (See 7.1.3.)

Results and Discussion

One laboratory reported single results for three samples: mean 66.5, and standard deviation 2.0. The other laboratory reported single results for two samples: mean 71.4, and standard deviation could not be determined.

Conclusions

In view of the large imprecision in the results, Zr concentrations determined by ICP may be expected to have large uncertainties, and its application for Zr in yttria-zirconia appears questionable. However, the amount of data was insufficient for an adequate analysis.

7.5. METHODS FOR YTTRIUM IN YTTRIA-ZIRCONIA

The largest number of laboratories reported results from ICP measurements. Two laboratories reported a total of nine determinations by XRF. AAS, neutron activation analysis and DCP also were used, but the amount of data was insufficient for statistical evaluation.

Data are summarized in Section 7.6.5.

7.5.1. Neutron Activation Analysis (NAA)

Use and Significance

The utility of neutron activation analysis is limited by the availability of a fission reactor facility and the turn-around time for analysis. Consequently, NAA is usually applied selectively when the value of the material is important, when the material cannot be analyzed accurately by other methods, and when high accuracy or low trace level limits are important. The method (thermal neutrons) is not recommended for light elements C, N, O, F, Si, P, Al, Mg and S, as well as Fe, due to their low cross-sections for thermal neutrons. Fast neutron activation is required for light elements.

Results and Discussion

One determination of Y concentration by NAA was reported with a value of 4.30 percent by weight.

Conclusions

Data were insufficient to establish between-laboratory and sample-to-sample variances. Due to the expense of this method, detailed testing is recommended only for those cases where other methods prove inadequate.

7.5.2. X-ray Fluorescence (See 7.1.1.)

Instrument and Experimental Parameters

One laboratory dissolved their samples by fusion in lithium tetraborate. A second laboratory reported dispersion of the powder in a special wax matrix.

Results and Discussion

The combined results for the two laboratories are summarized in Table 7.4.1.1. The values obtained using the lithium tetraborate flux were slightly less than the value obtained by NAA; while those obtained using the wax matrix were slightly above. However, due to the small amount of data, a high degree of confidence cannot be assigned to these apparent biases.

Conclusions

Results are consistent with NAA. Additional data for interlaboratory comparison of this method are desirable toward its possible establishment as a reference method for yttria-zirconia.

7.5.3. Atomic Absorption Spectrometry (See 7.1.2.)

Results and Discussion

One laboratory reported single results for three samples: mean 4.46, and standard deviation 0.11. The precision appears comparable to that obtained with XRF, but a between-laboratory component was not included in the present result.

Conclusions

Data were insufficient for a complete analysis of variance.

7.5.4. Inductively Coupled Plasma (See 7.1.3.)

Results and Discussion

Single determinations for seven samples were reported by 5 laboratories: mean 4.16, standard deviation 0.43.

Conclusions

In view of the large uncertainty associated with this method, the accuracy of the method or the reliable procedures may be suspected for analysis of yttrium

7.5.5. Direct-Current Plasma (See 7.1.4.)

Results and Discussion

Two results reported by two laboratories had a mean 4.3 ± 0.1 .

Conclusions

Data were insufficient for an analysis of variance. However, based on very limited results, the observed bias and imprecision obtained with this method suggest that this method should not be used for accurate or precise work.

7.6. MAJOR CHEMICAL COMPONENTS - STATISTICAL DATA

7.6.1. Silicon in Silicon Nitride, Silicon Carbide and Silicon

7.6.1.1 X-ray Fluorescence Spectrometry

Table 7.6.1.1.1 Silicon Concentration (wt. %) in SNR Powder

Material: Silicon Nitride, Reference Powder

Property: Si

Method: 12

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
5	1	59.90	59.90	59.90	0.00
25	1	59.40	59.40	59.40	0.00
Means	2	59.40	59.90	59.65	0.35

Table 7.6.1.1.2 Silicon Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder

Property: Si

Method: 12

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
5	6	59.60	59.90	59.75	0.10
25	6	59.18	59.43	59.31	0.10
Means	2	59.31	59.75	59.53	0.30

Table 7.6.1.1.3 Silicon Concentration (wt. %) in SiC Powder

Material: Silicon Carbide
 Property: Si
 Method: 12

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
25	6	69.11	69.78	69.47	0.23
Means	1	69.47	69.47	69.47	0.00

7.6.1.2. Atomic Absorption Spectroscopy

Table 7.6.1.2.1 Silicon Concentration (wt. %) in SNR Powder

Material: Silicon Nitride, Reference Powder
 Property: Si
 Method: 1

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
14	2	59.50	59.50	59.50	0.00
Means	1	59.50	59.50	59.50	0.00

Table 7.6.1.2.2 Silicon Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder
 Property: Si
 Method: 1

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
14	1	60.00	60.00	60.00	0.00
Means	1	60.00	60.00	60.00	0.00

Table 7.6.1.2.3 Silicon Concentration (wt. %) in Si Powder

Material: Silicon

Property: Si

Method: 1

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	1	99.40	99.40	99.40	0.00
Means	1	99.40	99.40	99.40	0.00

7.6.1.3. Inductively Coupled Plasma - Emission Spectroscopy

Table 7.6.1.3.1 Silicon Concentration (wt. %) in SNR Powder

Material: Silicon Nitride, Reference Powder

Property: Si

Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	2	56.30	58.50	57.40	1.55
6	1	58.07	58.07	58.07	0.00
15	1	59.38	59.38	59.38	0.00
21	4	58.68	59.92	59.42	0.53
Means	4	57.40	59.42	58.56	0.99

Table 7.6.1.3.2 Silicon Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder (Figure 67)
 Property: Si
 Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
6	1	59.10	59.10	59.10	0.00
15	1	60.15	60.15	60.15	0.00
21	4	59.60	60.40	59.97	0.33
Means	3	59.10	60.15	59.74	0.56

Table 7.6.1.3.3 Silicon Concentration (wt. %) in SiC Powder

Material: Silicon Carbide
 Property: Si
 Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
6	1	63.73	63.73	63.73	0.00
15	1	70.11	70.11	70.11	0.00
21	3	69.00	69.30	69.16	0.15
Means	3	63.73	70.11	67.66	3.44

Table 7.6.1.3.4 Silicon Concentration (wt. %) in Si Powder

Material: Silicon
 Property: Si
 Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
6	1	94.06	94.06	94.06	0.00
15	1	98.59	98.59	98.59	0.00
Means	2	94.06	98.59	96.32	3.20

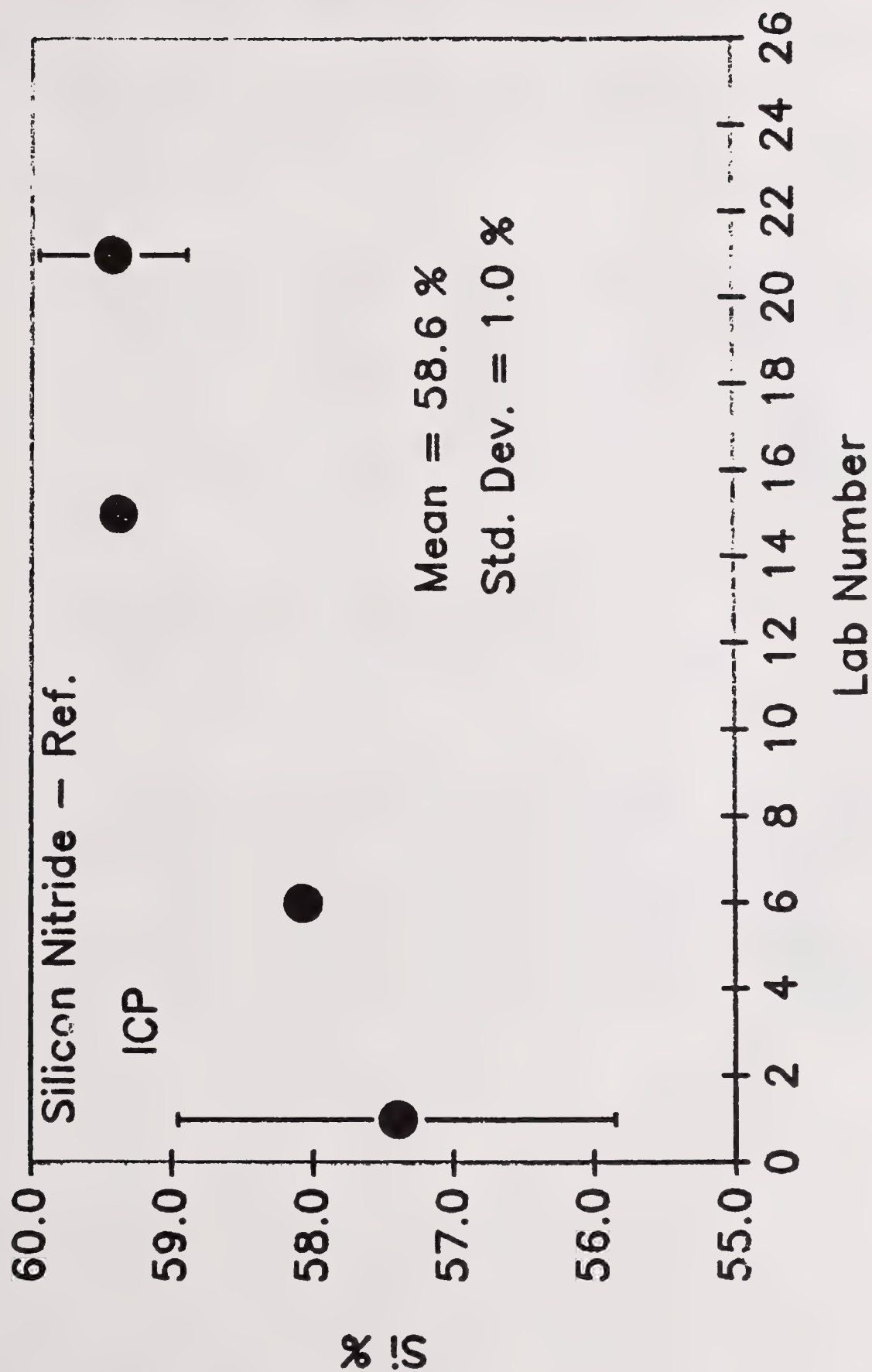


Figure 67. Silicon concentration (wt. %) in SNR powder as reported by participating labs (identified by number) for ICP Analysis

7.6.1.4. DC Plasma - Emission Spectroscopy

Table 7.6.1.4.1 Silicon Concentration (wt. %) in SNR Powder

Material: Silicon Nitride, Reference Powder
Property: Si
Method: 17

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	6	57.40	61.40	59.10	1.54
3	1	59.10	59.10	59.10	0.00
18	1	59.41	59.41	59.41	0.00
Means	3	59.10	59.41	59.20	0.17

Table 7.6.1.4.2 Silicon Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder
Property: Si
Method: 17

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	2	61.60	62.60	62.10	0.70
18	1	58.89	58.89	58.89	0.00
Means	2	58.89	62.10	60.49	2.26

7.6.1.5. Gravimetry

Table 7.6.1.5.1 Silicon Concentration (wt. %) in SNR Powder

Material: Silicon Nitride, Reference Powder

Property: Si

Method: 20

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
13	11	59.05	59.76	59.47	0.19
18	1	58.74	58.74	58.74	0.00
24	3	58.10	60.20	59.43	1.15
Means	3	58.74	59.47	59.21	0.41

Table 7.6.1.5.2 Silicon Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder

Property: Si

Method: 20

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
18	1	59.46	59.46	59.46	0.00
24	2	59.10	59.40	59.25	0.21
Means	2	59.25	59.46	59.35	0.14

Table 7.6.1.5.3 Silicon Concentration (wt. %) in SiC Powder

Material: Silicon Carbide

Property: Si

Method: 20

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
8	2	65.86	68.63	67.24	1.95
18	1	68.49	68.49	68.49	0.00
Means	2	67.24	68.49	67.86	0.88

Table 7.6.1.5.4 Silicon Concentration (wt. %) in Si Powder

Material: Silicon

Property: Si

Method: 20

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
5	6	98.60	98.90	98.80	0.10
8	2	94.77	98.19	96.48	2.41
18	1	99.00	99.00	99.00	0.00
Means	3	96.48	99.00	98.09	1.40

7.6.2 Nitrogen in Silicon Nitride

7.6.2.1 Combustion

Table 7.6.2.1.1 Nitrogen Concentration (wt. %) in SNR Powder

Material: Silicon Nitride, Reference Powder (Figure 68)

Property: N

Method: 8

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	6	38.33	38.67	38.50	0.13
3	8	38.20	38.30	38.22	0.04
5	1	38.50	38.50	38.50	0.00
6	4	37.70	38.50	38.10	0.40
13	11	40.03	41.52	40.79	0.48
14	1	38.00	38.00	38.00	0.00
18	2	32.10	37.58	34.84	3.87
19	1	37.72	37.72	37.72	0.00
21	3	38.10	38.30	38.20	0.10
24	3	38.30	40.50	39.36	1.10
Means	10	34.84	40.79	38.22	1.48

Table 7.6.2.1.2 Nitrogen Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder (Figure 69)

Property: N

Method: 8

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	4	37.40	38.40	38.00	0.45
5	4	38.40	39.30	38.90	0.39
6	2	38.50	39.00	38.75	0.35
14	1	38.50	38.50	38.50	0.00
15	1	38.10	38.10	38.10	0.00
18	1	32.30	32.30	32.30	0.00
19	7	37.88	39.20	38.43	0.43
21	6	38.70	38.90	38.83	0.08
24	14	38.50	41.00	39.65	0.73
Means	9	32.30	39.65	37.94	2.17

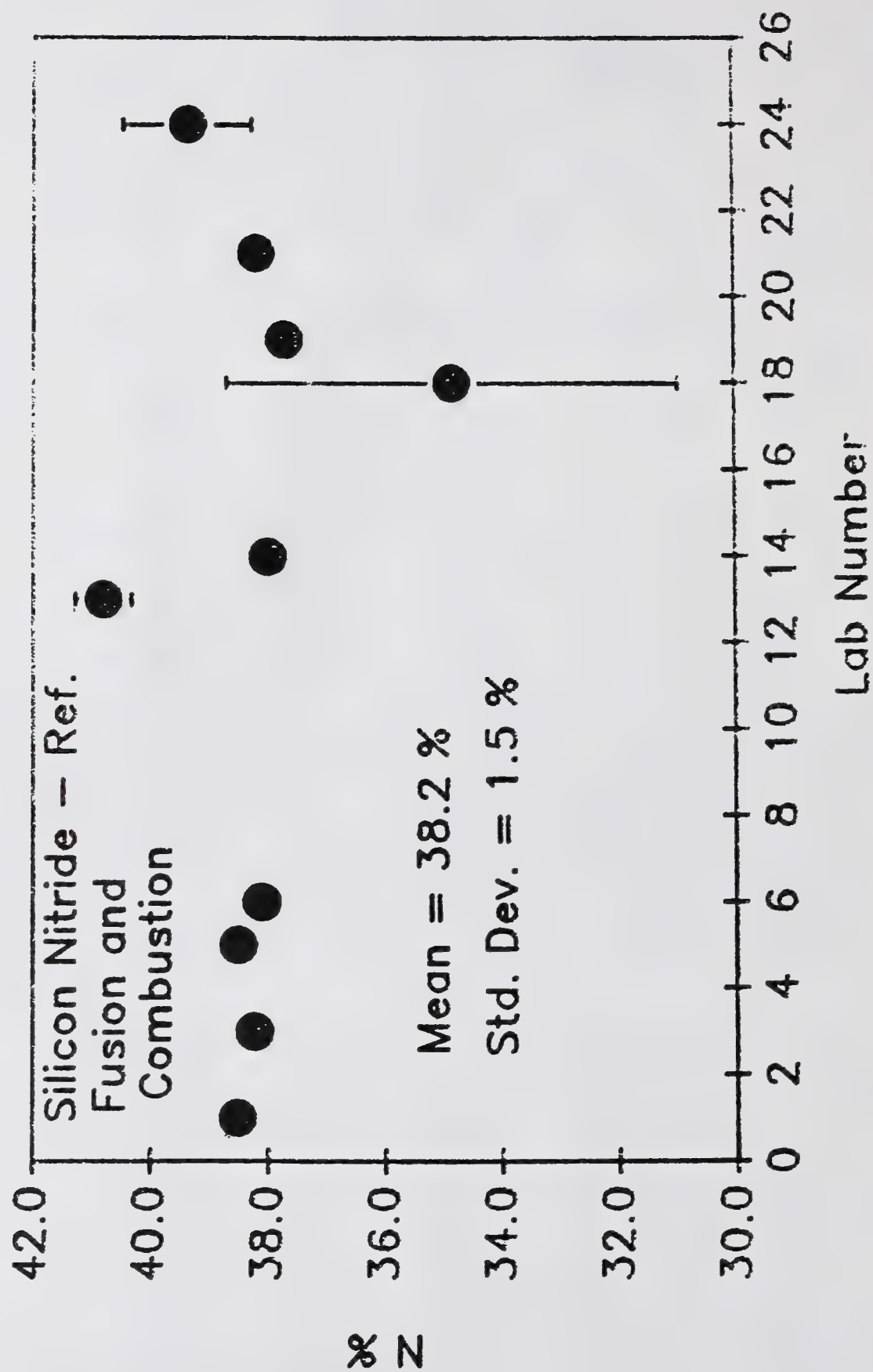


Figure 68. Nitrogen concentration (wt. %) in SNR powder as reported by labs using Combustion Analysis

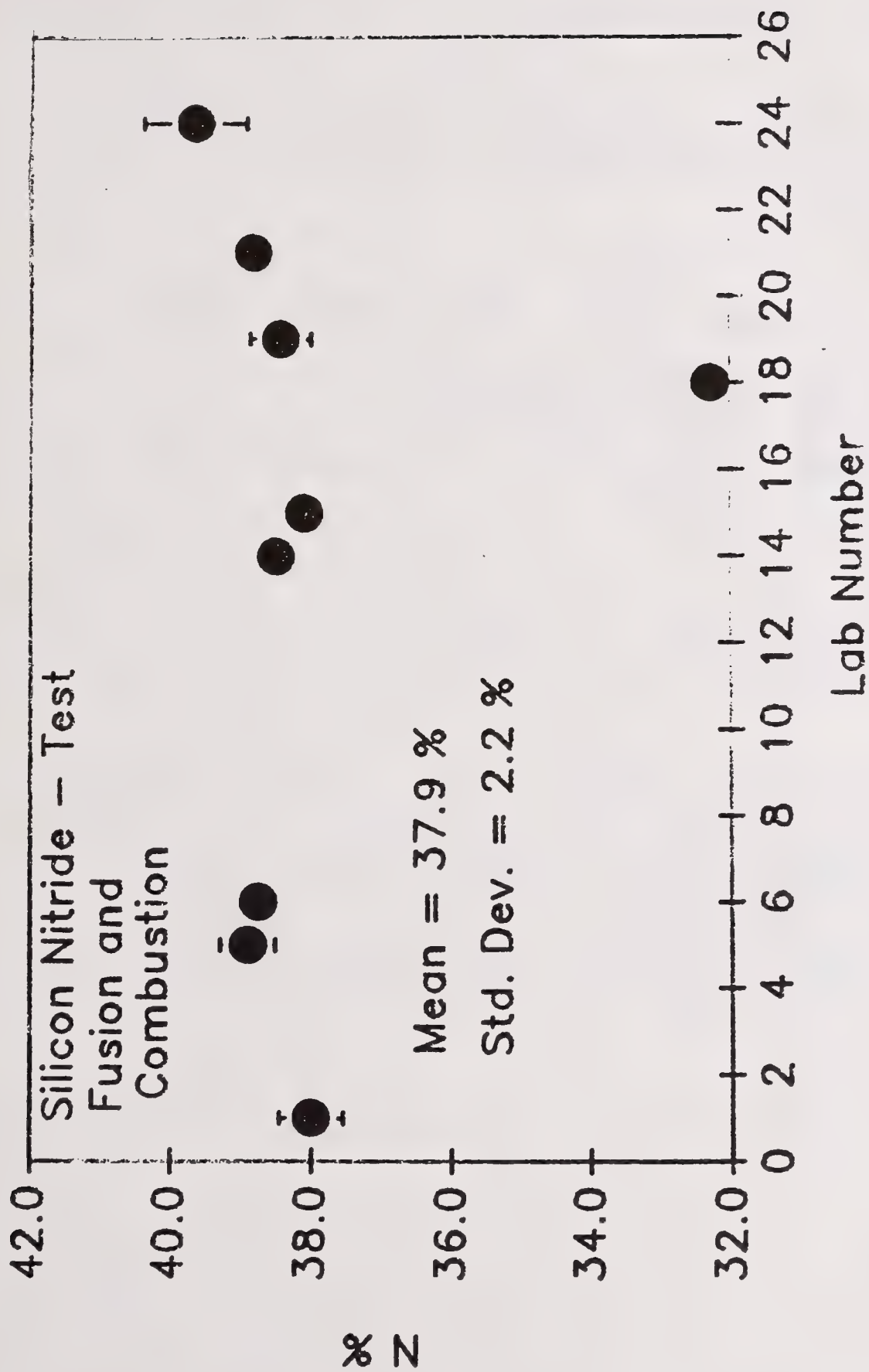


Figure 69. Nitrogen concentration (wt. %) in SNT powder as reported by labs using Combustion Analysis

7.6.2.2. Kjeldahl Analysis

Table 7.6.2.2.1 Nitrogen Concentration (wt. %) in SNR Powder

Material: Silicon Nitride, Reference Powder

Property: N

Method: 15

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
15	1	38.91	38.91	38.91	0.00
25	1	38.40	38.40	38.40	0.00
Means	2	38.40	38.91	38.65	0.36

Table 7.6.2.2.2 Nitrogen Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder

Property: N

Method: 15

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
5	4	38.70	39.10	38.87	0.17
15	1	39.04	39.04	39.04	0.00
25	6	38.79	39.00	38.91	0.08
Means	3	38.87	39.04	38.94	0.08

7.6.2.3 Potentiometric Titration

Table 7.6.2.3.1 Nitrogen Concentration (wt. %) in SNR Powder by Potentiometric Titration

Material: Silicon Nitride, Reference Powder

Property: N

Method: 13

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
24	3	32.07	40.50	36.72	4.28
Means	1	36.72	36.72	36.72	0.00

Table 7.6.2.3.2 Nitrogen Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder

Property: N

Method: 13

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
24	2	37.90	38.40	38.15	0.35
Means	1	38.15	38.15	38.15	0.00

7.6.3 Total Carbon in Silicon Carbide

7.6.3.1 Combustion

Table 7.6.3.1.1 Total Carbon Concentration (wt. %) in SiC Powder

Material: Silicon Carbide (Figure 70)

Property: C Total

Method: 8

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	4	28.89	29.01	28.95	0.05
6	2	31.45	31.46	31.45	0.00
7	2	30.10	30.10	30.10	0.00
8	2	28.96	29.45	29.20	0.34
15	1	29.34	29.34	29.34	0.00
18	1	29.70	29.70	29.70	0.00
Means	6	28.95	31.45	29.79	0.90

7.6.3.2. Electrochemical Methods - Potentiometric Titration

Table 7.6.3.2.1 Total Carbon Concentration (wt. %) in SiC Powder

Material: Silicon Carbide

Property: C Total

Method: 16

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
10	1	29.97	29.97	29.97	0.00
21	3	30.04	30.10	30.08	0.03
25	6	29.97	30.07	30.03	0.04
Means	3	29.97	30.08	30.02	0.05

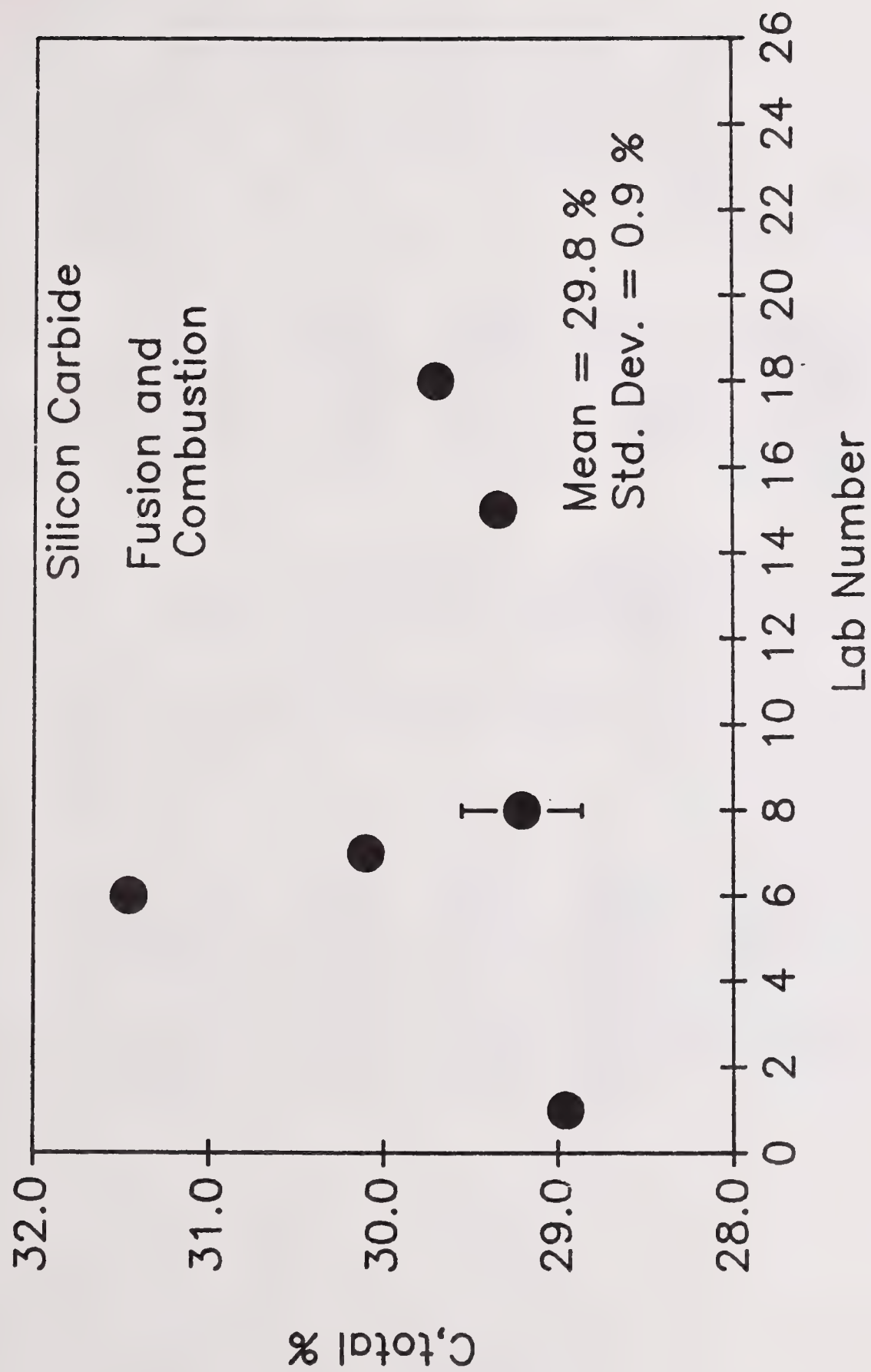


Figure 70. Total Carbon concentration (wt. %) in SiC powder as reported by labs using Combustion Analysis

7.6.3.3. Gravimetry

Table 7.6.3.3.1 Total Carbon Concentration (wt. %) in SiC Powder

Material: Silicon Carbide

Property: C Total

Method: 20

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
18	1	29.70	29.70	29.70	0.00
Means	1	29.70	29.70	29.70	0.00

7.6.4. Zirconium in Yttria-Zirconia

7.6.4.1 X-ray Fluorescence Spectrometry

Table 7.6.4.1.1 Zirconium Concentration (wt. %) in YSZ Powder

Material: Zirconia

Property: Zr

Method: 12

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
22	6	68.44	68.54	68.50	0.03
Means	1	68.50	68.50	68.50	0.00

7.6.4.2 Atomic Absorption Spectroscopy

Table 7.6.4.2.1 Zirconium Concentration (wt. %) in YSZ Powder

Material: Zirconia
Property: Zr
Method: 1

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
18	3	67.00	67.20	67.10	0.10
Means	1	67.10	67.10	67.10	0.00

7.6.4.3 Inductively Coupled Plasma - Emission Spectroscopy

Table 7.6.4.3.1 Zirconium Concentration (wt. %) in YSZ Powder

Material: Zirconia
Property: Zr
Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	2	65.00	65.60	65.30	0.42
6	1	68.78	68.78	68.78	0.00
22	2	71.40	71.40	71.40	0.00
Means	3	65.30	71.40	68.49	3.06

7.6.5. Yttrium in Yttria-Zirconia

7.6.5.1 Neutron Activation Analysis

Table 7.6.5.1.1 Yttrium Concentration (wt. %) in YSZ Powder

Material: Zirconia

Property: Y

Method: 6

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
2	1	4.30	4.30	4.30	0.00
Means	1	4.30	4.30	4.30	0.00

7.6.5.2 X-ray Fluorescence Spectrometry

Table 7.6.5.2.1 Yttrium Concentration (wt. %) in YSZ Powder

Material: Zirconia

Property: Y

Method: 12

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
22	6	4.15	4.23	4.18	0.02
24	3	4.52	4.52	4.52	0.00
Means	2	4.18	4.52	4.35	0.23

7.6.5.3 Atomic Absorption Spectroscopy

Table 7.6.5.3.1 Yttrium Concentration (wt. %) in YSZ Powder

Material: Zirconia (Figure 71)

Property: Y

Method: 1

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
18	3	4.33	4.54	4.45	0.11
Means	1	4.45	4.45	4.45	0.00

7.6.5.4 Inductively Coupled Plasma - Emission Spectroscopy

Table 7.6.5.4.1 Yttrium Concentration (wt. %) in YSZ Powder

Material: Zirconia

Property: Y

Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	2	3.88	3.92	3.90	0.02
6	1	3.48	3.48	3.48	0.00
15	1	4.11	4.11	4.11	0.00
17	1	4.52	4.52	4.52	0.00
22	2	4.60	4.60	4.60	0.00
Means	5	3.48	4.60	4.12	0.46

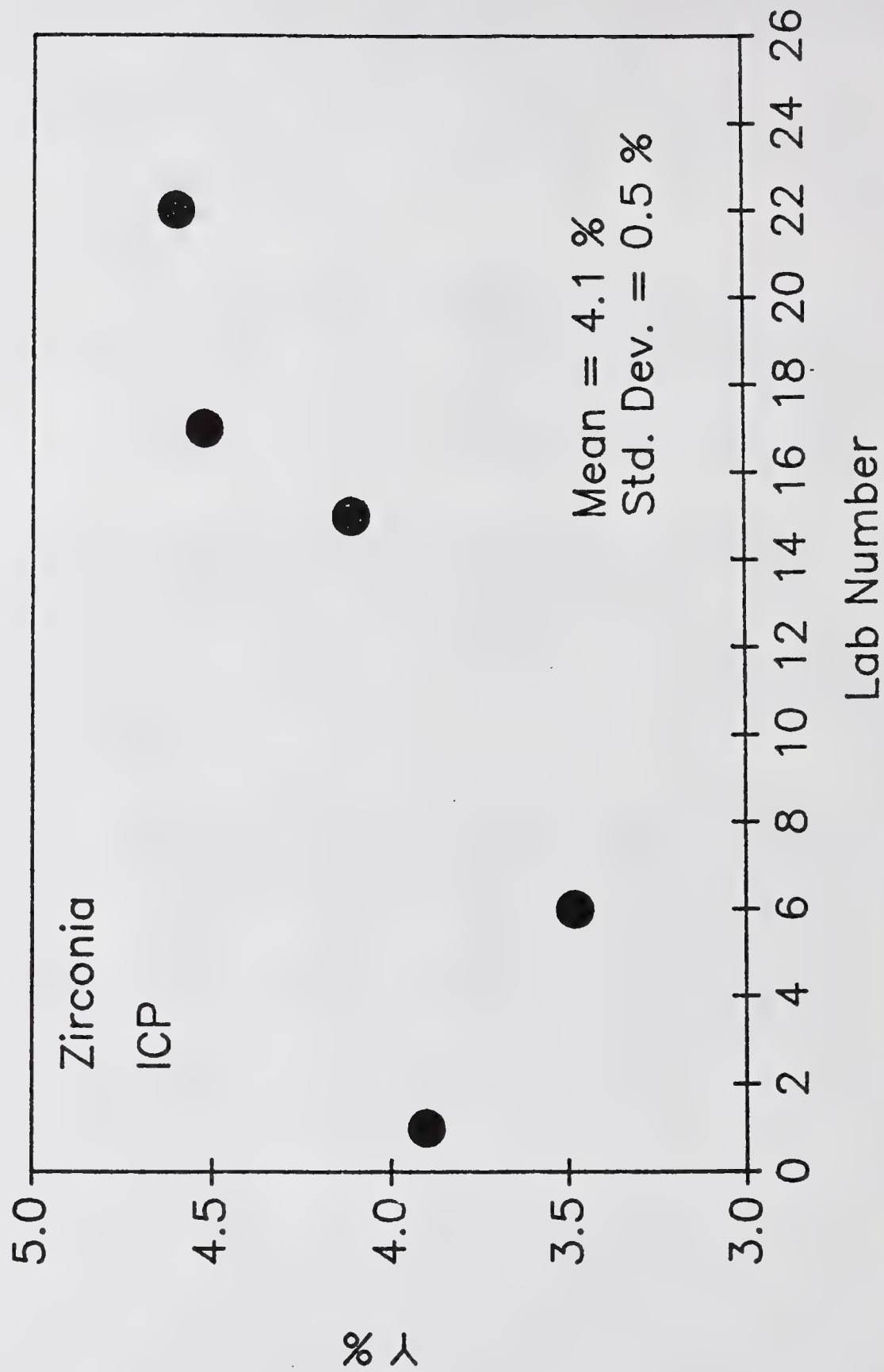


Figure 71. Yttrium concentration (wt. %) in YSZ powder as reported by labs using ICP-Emission Spectroscopy

7.6.5.5 DC Plasma - Emission Spectroscopy

Table 7.6.5.5.1 Yttrium Concentration (wt. %) in YSZ Powder

Material: Zirconia

Property: Y

Method: 17

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
14	1	4.20	4.20	4.20	0.00
17	1	4.41	4.41	4.41	0.00
Means	2	4.20	4.41	4.30	0.14

7.7. STATISTICAL ANALYSIS, R. Munro, NIST

Quantitative analysis of the major elemental components of the powders generally involved some form of decomposition of the material. Results from two methods, the combustion and Kjeldahl techniques which used chemical reactions to decompose silicon nitride, are shown in Figures 72-73 for the determination of nitrogen. The combustion method involves the detection and measurement of the quantity of gaseous nitrogen released from a measured mass of silicon nitride. The Kjeldahl method is a neutralization titration technique that determines the amount of nitrogen based on the amount of ammonia produced by a controlled chemical reaction.

The level of precision of the Kjeldahl method was only somewhat better than the precision attained by the combustion technique. The between-laboratory variances, however, were significantly different. Much better agreement between laboratories was achieved by the Kjeldahl method.

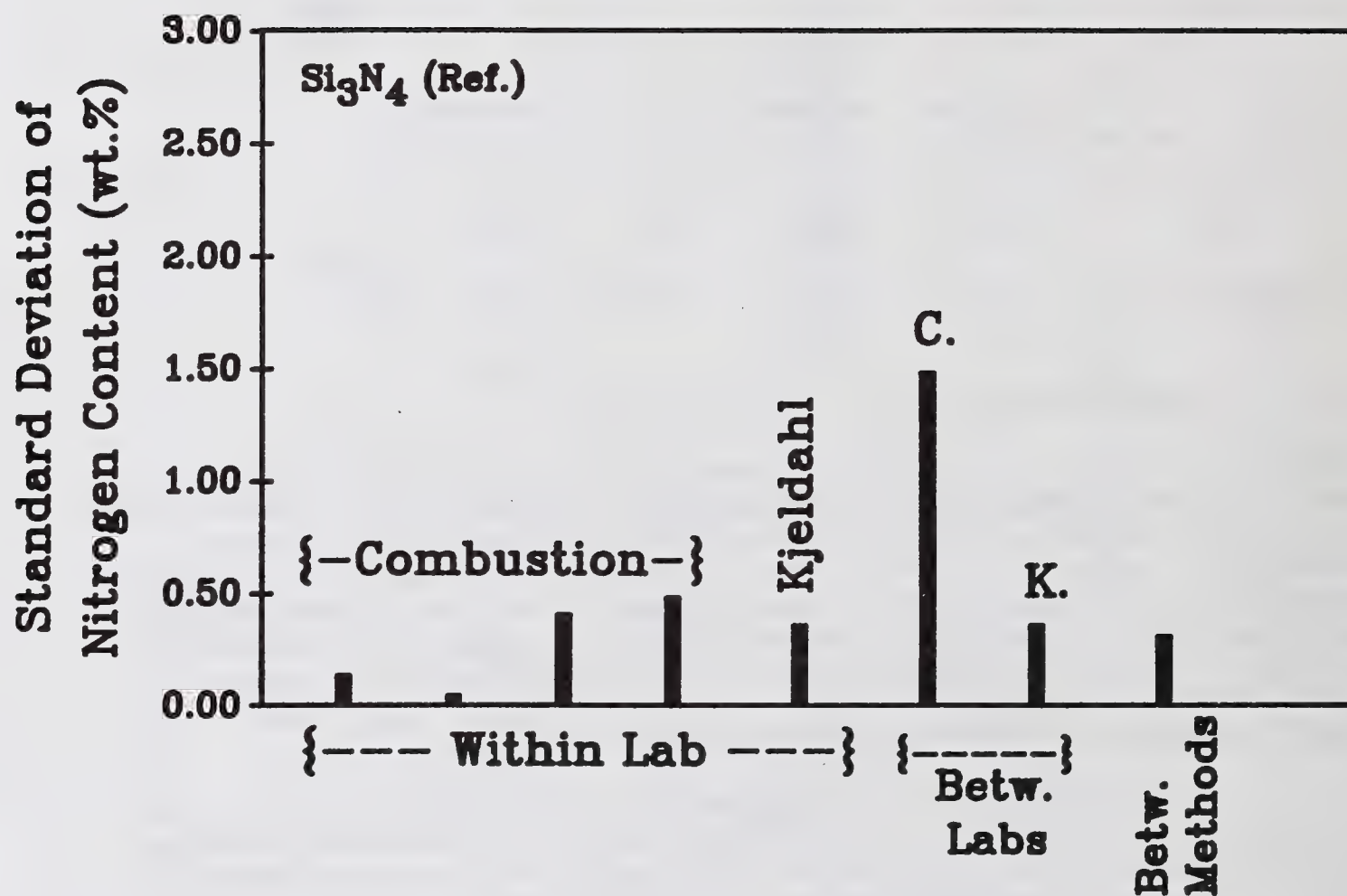


Figure 72. Within lab, between lab, and between methods standard deviations in the measurement of nitrogen content for the Si_3N_4 (Ref.) powder as measured by various laboratories using a variety of methods.

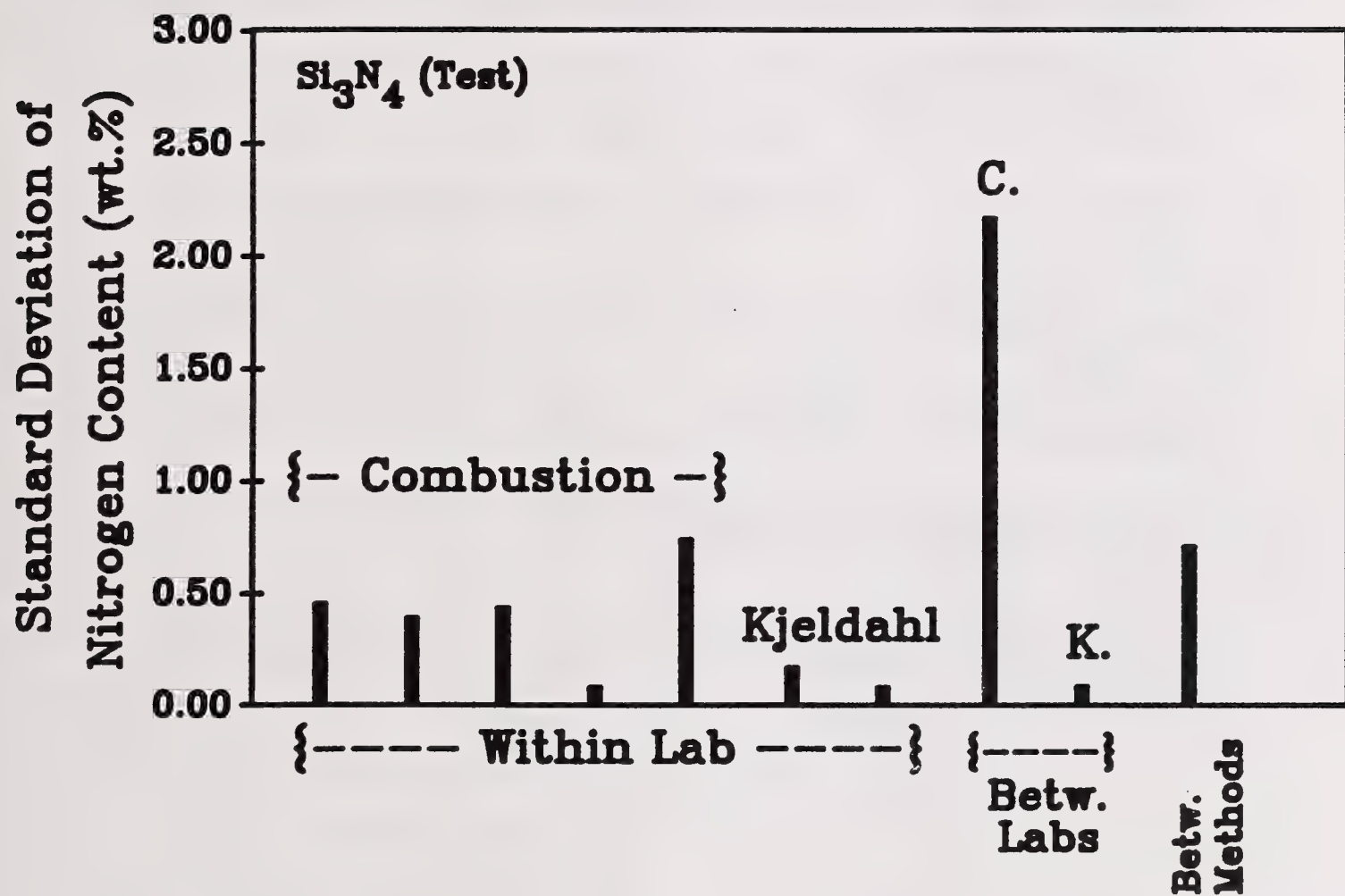


Figure 73. Within lab, between lab, and between methods standard deviations in the measurement of nitrogen content for the Si₃N₄(Test) powder as measured by various laboratories using a variety of methods.

7.8 References

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8. MAJOR NONMETALLIC IMPURITIES (Concentration > 100 ppm by weight)

A. L. Dragoo, S. G. Malghan, R. G. Munro and S. M. Hsu; NIST

8.1. METHODS FOR TOTAL CARBON IN SILICON NITRIDE AND SILICON

Combustion analysis was the principal method used for determination of carbon impurity in silicon nitride and silicon. Other decomposition methods employed electrochemical measurements and gravimetric analysis; however, the amount of data reported was insufficient for evaluation of these other methods.

Data are summarized in Section 8.10.2.

8.1.1. Combustion (See 7.2.1.)

Results and Discussion

The results are summarized in Table 7.3.1.1 for SNR Si_3N_4 , SNT Si_3N_4 and Si. The method of detection was presumed to be IR absorption in all cases. The standard deviations for the total set of measurements for each material are comparable to the within laboratory standard deviation for that material.

The results for determination of total carbon impurity by combustion analysis are compared in Table 8.1.2.1 with those obtained by electrochemical methods and gravimetry.

Conclusions

Good agreement was evident among laboratories using combustion methods. Presently, lower limit of reliable detectability for carbon impurity in Si_3N_4 and Si by combustion analysis appears to be about 0.05%.

8.1.2. Electrochemical Methods (See 7.2.)

Instrument and Experimental Parameters

Potentiometry

The experimental method (Lab 24) was comparable to the combustion method; however, the evolved CO_2 was collected in $\text{Ba}(\text{OH})_2$ solution and the solution was potentiometrically titrated to determine the amount of CO_2 recovered.

Coulometry

Two methods were reported.

The first method was used by Lab 3. A powder sample was decomposed in a $\text{PbO/B}_2\text{O}_3$ flux and the carbon reacted with a stream of O_2 . The CO_2 was recovered by precipitation of BaCO_3 . The amount of BaCO_3 formed was determined by coulometric titration to a constant pH (pH = 10).

The second method was used by Lab 21. The sample was oxidized with O₂, the CO₂ recovered (procedure not specified) and the amount determined by coulometric titration.

Results and Discussion

As shown by the results in Table 8.1.2.1, coulometric titration appears to have yielded results in agreement with those obtained by combustion followed by IR detection; whereas, the results obtained by potentiometric titration appear to be low. Since both electrochemical methods employ combustion of the sample to obtain the analyte, it is expected that recovery of CO₂ may have been incomplete in the case of the potentiometric titration method.

The precision of the two electrochemical methods appears to be comparable.

Conclusions

Results obtained with potentiometric titration may have been low due to insufficient recovery of the evolved CO₂ from the decomposition process. However, data were insufficient for a complete evaluation of the method.

Table 8.1.2.1 Comparison of Methods of Determination of Total Carbon Impurity in Silicon Nitride and Silicon Powder Samples

Material	Method	Number of		Mean	Standard Deviation
		Labs	Samples		
SNR	Combustion	13	22	0.15	0.02
SNR	Potentiometric	1	3	0.04	0.01
SNR	Coulometric	2	5	0.29	0.03
SNT	Combustion	12	23	0.13	0.03
SNT	Potentiometric	1	2	0.053	0.009
SNT	Coulometric	1	1	0.11	0.01
SNT	Gravimetry	1	1	0.12	--
Si	Combustion	8	12	0.08	0.03
Si	Coulometric	1	4	0.061	0.002

8.2. METHODS FOR FREE CARBON IN SILICON CARBIDE

A very limited number of determinations of free-carbon in silicon carbide were reported by combustion analysis, coulometric titration and gravimetric analysis. Significant differences appear to exist in the data, and the data were insufficient for statistical evaluation.

Data are summarized in Section 8.10.3.

8.2.1. Combustion (See 7.2.1.)

Instrument and Experimental Parameters

A standard method for determination of free carbon in SiC by combustion analysis is described in DIN 51075, Part 2. Kerber et al.⁹ discussed the problems encountered in applying this method to fine SiC powders.

The sample was treated in an oxidizing atmosphere to convert the free carbon to CO₂. The amount of CO₂ generated was measured by IR absorption.

8.3. METHODS FOR TOTAL CARBON IN YTTRIA-ZIRCONIA

Only combustion analysis was used to measure carbon in the yttria-zirconia powder.

Data are summarized in Section 8.10.3.

8.3.1. Combustion (See 7.2.1.)

Instrument and Experimental Parameters

A tin-coated Cu as an accelerator for combustion was used in the decomposition reaction. Measurements were calibrated with standards supplied by the vendor of the instrument.

Results and Discussion

The average result for the method is compared in Table 7.3.1.1 with results for combustion analysis of the other powders. Good agreement was evident between the three laboratories reporting. The improved sensitivity of this method for C in yttria-zirconia is to be noted as compared to similar determinations of C in Si₃N₄ and Si.

Conclusions

Combustion appears to offer a method for reproducible determination of C in yttria-zirconia.

8.4. METHODS FOR NITROGEN IN SiC, SILICON AND YTTRIA-ZIRCONIA

Nitrogen impurity concentrations were determined by combustion analysis.

Data are summarized in Section 8.10.5.

8.4.1. Combustion (See 7.2.1.)

Instrument and Experimental Parameters

SiC, Si: One laboratory reported that the samples were reacted with tin powder in a nickel basket. The same laboratory reported that standards supplied by the vendor of the instrument were used.

Results and Discussion

The results are summarized in Table 7.1.2.1. Good agreement is evident for results obtained for N in SiC. In the case of Si, the poorer precision makes the reproducibility of the measurements suspect. The lack of sufficient data does not permit the method to be adequately evaluated for this material.

Conclusions

Combustion analysis appears to offer good reproducibility and sensitivity for the determination of N in SiC. In the case of Si, the utility of this method is questionable in view of the low N concentration in the material and the apparent limiting precision of the method. Further evaluation of the method is required for yttria-zirconia.

8.5. METHODS FOR OXYGEN IN SILICON NITRIDE, SILICON CARBIDE AND SILICON

Oxygen impurity concentrations in these materials was carried out by a large number of laboratories using combustion analysis. In addition, the following four methods were used for oxygen analysis in silicon nitride, silicon carbide and silicon: fast neutron activation analysis, thermal neutron activation analysis, ICP and Coulometric titration.

Data are summarized in Section 8.10.6.

8.5.1. Fast Neutron Activation Analysis (FNAA)

Use and Significance

Fast neutron activation analysis is used for light elements where NAA with thermalized ("slow") neutrons is not indicated due to the low absorption cross-sections of light elements for thermal neutrons. Fast neutrons are used to produce (n,2n), (n,p) and (n, α) reactions. Results are given here for the analysis of oxygen in the four nonoxide powders. See also remarks in Section 7.5.1. on NAA.

Instrument and Experimental Parameters

Fast neutrons of different energies may be produced in several ways, but the most favorable energy for the analysis of light elements is 14 MeV. Monoenergetic neutrons of this energy can be produced by bombardment of a tritium target with deuterium ions to produce neutrons from a helium fusion reaction, ${}^3\text{H}(\text{d},\text{n}){}^4\text{H}$. Oxygen determination is carried out using the reaction ${}^{16}\text{O}(\text{n},\text{p}){}^{16}\text{N}$ with a threshold of 10 MeV. Fast neutron fluxes of about 2×10^8 neutrons/cm²/s are delivered to the sample, giving a detection limit of about 15 ppm for a 20 g sample. Time-dependent flux changes are monitored via a plastic scintillator placed behind the sample and coupled to a photomultiplier detector. The photomultiplier output is fed to a discriminator adjusted so that only pulses originating from a proton recoil of at least 7 MeV are recorded. The measurement is calibrated by analyzing oxygen standards along with unknowns. The use of benzoic acid, hydroxytriphenylsilane and N-1-naphthylacetamide as standards was reported in this study.

Samples were placed in small polyethylene vials with tight fitting caps and submitted along with standards for analysis. Results were uncorrected for fluorine which is a direct interference to the determination of oxygen. The correction for fluorine is:

$$\%O_{\text{corrected}} = \%O_{\text{expected}} - 0.4(\%F) \quad .$$

Results and Discussion

Results are summarized in Table 8.5.1.1. Although FNAA results are reported by only one of the participating laboratories, the results were obtained by submitting samples to laboratories specializing in FNAA. Since no difference was discernable in the results from both the FNAA laboratories, the results have been aggregated here.

One other laboratory reported results obtained with slow neutron activation. These are included for comparison in Table 8.5.1.1. The results obtained with FNAA for silicon nitride and SiC powders are in agreement with selected results obtained by combustion analysis: compare the results in Table 8.5.1.1 with those in Table 8.5.1.2.

The results obtained for O in Si are about 20% larger than those obtained by combustion analysis. The sources of this bias are not evident.

Conclusions

FNAA appears capable of providing accurate and precise data regarding the O-content of silicon nitride, SiC and Si. The utility of this method is limited by the requirement of a specialized facility. However, it is recommended that the services of one or more of such facilities should be employed in establishing an oxygen reference standard for nonoxide ceramics.

Table 8.5.1.1 Determination of O in Silicon Nitride, Silicon Carbide and Silicon by Neutron Activation Analysis. Concentrations are in percent by weight. "SLOW" refers to thermal neutrons.

MATERIAL	TYPE	NUMBER OF		MEAN	STANDARD DEVIATION
		LABS	SAMPLES		
SNR	FAST ^a	1	12	2.16	0.14
SNT	FAST	1	8	1.16	0.06
SiC	FAST	1	8	0.75	0.02
	SLOW	1	2	1.48	0.15
Si	FAST	1	8	0.64	0.04
	SLOW	1	2	1.70	0.18

^aCorrected for F.

8.5.2. Combustion (See 7.2.1.)

Instrument and Experimental Parameters

Oxygen was determined by measuring the thermal conductivity (TC) of the evolved gas, as with the case of N₂ above, or by recovering the oxygen in CO₂ and measuring the intensity of IR-absorption.

Samples were encapsulated in a metal, such as tin, with powdered nickel added as a flux. Reduction was carried out in a graphite crucible with helium or argon used as a carrier gas. Temperature and heating rate for the reaction may have significant effect on the extent of the reduction or on possible loss of oxygen as SiO. Purity of carrier gas is also important.

Results and Discussion

For SNR Si₃N₄, a total of 17 laboratories reported determinations of oxygen concentrations for 59 samples and 87 determinations. Six of the laboratories reporting results by TC detection and six reporting results by IR detection showed very good agreement, with means close to 2.22. One each of the labs reporting results by these two methods of detection reported significantly larger values: above 2.50. These higher values may be due to bias in experimental conditions or instrument calibrations since similar deviations were evident for SNT samples reported by the same laboratories.

Generally good agreement was found between the two methods of detection for SNT, SiC and Si samples.

Conclusions

Determination of oxygen-concentrations in silicon nitride, silicon carbide and silicon generally showed good agreement, although some cases of experimental bias were evident in the case of silicon nitride. Better definition of analytical conditions and development of a generally available reference material are recommended.

Table 8.5.2.1 Determination of O in Silicon Nitride, Silicon Carbide and Silicon by Combustion. Concentrations are in percent by weight.

MATERIAL	DETECTION	NUMBER OF		MEAN	STANDARD DEVIATION
		LABS.	SAMPLES		
SNR	TC	6	20	2.22	0.08
	TC	1	2	2.53	0.05
	IR	6	19	2.21	0.19
	IR	1	3	2.60	0.09
	NS ^a	3	5	2.14	0.07
SNT	TC	4	12	1.26	0.05
	IR	5	6	1.18	0.05
	IR	1	1	1.34	0.03
	NS	2	3	1.15	0.11
SiC	TC	1	2	0.64	--
	IR	4	5	0.72	0.07
	NS	1	1	0.74	0.01
Si	TC	3	4	0.51	0.01
	IR	4	8	0.53	0.02
	NS	1	1	0.50	0.04

^aNS, method of detection not specified.

8.6. METHODS FOR FLUORINE IN SILICON NITRIDE AND SILICON CARBIDE

Fluorine impurity concentrations in silicon nitride and silicon carbide were measured by mass spectrometry, ion chromatography, potentiometric titration and specific ion electrode. The amount of data reported for each method was insufficient for an adequate evaluation of the method.

8.6.1. Ion Chromatography (IC)

Use and Significance

Ion chromatography is a highly efficient method for the determination of anions including F^- , Cl^- , and SO_4^{2-} ; especially at low concentrations of these ions. A prerequisite for the use of this method is the dissolution of the powder in an appropriate solvent.

Instrument and Experimental Parameters

The three major components of an ion chromatography system are the separator columns, suppressor columns and conductivity detectors. Some of the most common separator columns are AS-1 and AS-2^(R), which have polystyrene/divinylbenzene base resin core particles with a diameter of 25 μm . A common eluent is bicarbonate/carbonate for common anion chromatography. The suppressor column in AS-1 converts bicarbonate/carbonate eluent to carbonic acid. Since the carbonic acid has a high conductivity, it increases the detection limit. However, in AS-3 the suppressor column consists of potassium phosphate.

No information was given in this study with regard to the types of columns and eluents used.

Depending on the powder composition and laboratory practice, different procedures are used for powder dissolution or for powder leaching. One laboratory reported that F^- was extracted by leaching with water (water temperature unreported). Details were not given for the ion chromatography measurement.

Measurements may be affected by filtration and preconcentration of dilute samples or by dilution of highly concentrated samples.

Results and Discussion

Results are comparable to those obtained by electrochemical methods (selective ion electrode and potentiometric titration). Measurements were not reported for the SiC powder.

(R) - Registered name by Dionics Corporation

8.6.2 Electrochemical Methods (See 7.2.3.)

Experimental Parameters

Specific Ion Electrode (SIE)

Experimental details were not reported.

Potentiometric Titration

In the case of the silicon nitride powders, one laboratory reported that analytical samples were dissolved in a NaOH-NaO₂ flux, followed by dissolution of the material in sulphuric acid. Experimental details were not given by the other two laboratories.

Results and Discussion

Average results of these two methods are consistent with each other and with IC in the cases of the two silicon nitride powders. However, data are insufficient for a reliable evaluation of the uncertainties of these methods.

8.7. METHODS FOR CHLORINE IN SILICON CARBIDE AND SILICON

Chlorine concentrations were determined in silicon carbide and silicon by ion chromatography - one determination on one sample for each material. Although the reported values were in excess of 100 ppm, the results are included in Section 10.5 for comparison with those for the two silicon nitride powders.

8.8. METHODS FOR SULFUR IN YTTRIA-ZIRCONIA

Results of a few measurements of sulfur concentrations were reported for ICP, mass spectrometry and combustion analysis. Data were insufficient for a reliable evaluation and comparison of methods.

Data are summarized in Section 8.10.8.

8.8.1 Ion Chromatography (See 7.3.1.)

Instrument and Experimental Parameters

One laboratory reported the extraction of leachable anions with boiling water.

Results and Discussion

Results are low relative to those obtained by combustion. The source of this discrepancy cannot be established from the data reported. However, it is to be noted that the combustion method would recover S from both the surface and interior of the particles; whereas, a leaching method may have extracted only S at or near the surface of the particles.

Conclusions

Results are low as noted above. This method may be more appropriate for surface, rather than bulk, analysis of powders.

8.9. METHODS FOR CHLORINE IN YTTRIA-ZIRCONIA

A few measurements of chlorine concentrations in yttria-zirconia were made by ion chromatography, neutron activation analysis and argentometric titration. The data were insufficient for an evaluation and comparison of these methods.

Data are summarized in Section 8.10.9.

8.10. STATISTICAL DATA ON MAJOR NONMETALLIC IMPURITIES (concentration > 100 ppm by weight)

Impurities present at concentrations greater than 100 ppm are termed "major impurities." This lower boundary for the major impurity category is arbitrary, and in a few instances it was desirable to include cases in which the impurity concentration was less than 100 ppm for a particular material because it permitted a convenient comparison with another material in which the impurity concentration was greater than 100 ppm. For example, see Tables 8.10.7.2 and 3 for the impurity concentration of F in the SNR and SNT powders, respectively.

8.10.1. Free Silicon in Silicon Nitride

8.10.1.1 Electrochemical Methods - Potentiometric Titration

Table 8.10.1.1 Free Silicon Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder
Property: FREESI
Method: 4

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
16	5	0.021	0.029	0.02	0.004
Means	1	0.025	0.025	0.02	0.00

8.10.1.2 Volumetric Determination

Table 8.10.1.2 Free Silicon Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder
 Property: FREESI
 Method: 9

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
21	2	0.07	0.09	0.08	0.014
Means	1	0.08	0.08	0.08	0.00

8.10.1.3 Standardized Method (DIN 51075)

Table 8.10.1.3 Free Silicon Concentration (wt. %) in SNR Powder

Material: Silicon Nitride, Reference Powder
 Property: FREESI
 Method: 18

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
3	4	0.1000	0.1400	0.1250	0.0173
Means	1	0.1250	0.1250	0.1250	0.0000

8.10.2 Total Carbon in Silicon Nitride and Silicon

8.10.2.1 Combustion Analysis

Table 8.10.2.1.1 Carbon Concentration (wt. %) in SNR Powder

Material: Silicon Nitride, Reference Powder

Property: C Total

Method: 8

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	6	0.15	0.17	0.16	0.00
5	1	0.12	0.12	0.12	0.00
6	5	0.14	0.16	0.15	0.00
7	17	0.11	0.17	0.14	0.01
14	2	0.16	0.16	0.16	0.00
15	1	0.15	0.15	0.15	0.00
16	4	0.15	0.17	0.15	0.00
17	2	0.15	0.19	0.17	0.02
18	3	0.13	0.16	0.14	0.01
19	1	0.19	0.19	0.19	0.00
22	10	0.15	0.18	0.16	0.01
24	1	0.17	0.17	0.17	0.00
25	1	0.18	0.18	0.18	0.00
Means	13	0.12	0.19	0.15	0.01

Table 8.10.2.2.2 Carbon Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder

Property: C Total

Method: 8

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	4	0.10	0.16	0.13	0.03
5	4	0.09	0.10	0.09	0.00
6	3	0.09	0.09	0.09	0.00
7	5	0.19	0.22	0.20	0.01
14	1	0.10	0.10	0.10	0.00
15	1	0.10	0.10	0.10	0.00
16	12	0.11	0.16	0.13	0.01
18	2	0.12	0.16	0.14	0.02
19	7	0.11	0.16	0.12	0.01
22	6	0.11	0.14	0.12	0.01
24	8	0.10	0.14	0.11	0.01
25	6	0.12	0.14	0.13	0.00
Means	12	0.09	0.20	0.12	0.02

Table 8.10.2.2.3 Carbon Concentration (wt. %) in Si Powder

Material: Silicon

Property: C Total

Method: 8

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	2	0.08	0.08	0.08	0.00
5	4	0.05	0.05	0.05	0.00
6	3	0.08	0.08	0.08	0.00
7	2	0.07	0.07	0.07	0.00
8	2	0.01	0.02	0.02	0.00
15	1	0.05	0.05	0.05	0.00
16	9	0.08	0.12	0.09	0.01
18	1	0.21	0.21	0.21	0.00
22	6	0.06	0.07	0.07	0.00
Means	9	0.02	0.21	0.08	0.05

8.10.2.2 Electrochemical Methods - Potentionmetric Titration

Table 8.10.2.2.1 Carbon Concentration (wt. %) in SNR Powder

Material: Silicon Nitride, Reference Powder
 Property: C Total
 Method: 13

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
24	4	0.0270	0.0520	0.0352	0.0114
Means	1	0.0352	0.0352	0.0352	0.0000

Table 8.10.2.2.2 Carbon Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder
 Property: C Total
 Method: 13

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
24	4	0.0450	0.0630	0.0532	0.0091
Means	1	0.0532	0.0532	0.0532	0.0000

8.10.2.3 Electrochemical Methods - Coulometric Titration

Table 8.10.2.3.1 Carbon Concentration (wt. %) in SNR Powder

Material: Silicon Nitride, Reference Powder
Property: C Total
Method: 16

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
3	4	0.2500	0.2800	0.2650	0.0129
21	3	0.3000	0.3300	0.3133	0.0153
Means	2	0.2650	0.3133	0.2892	0.0342

Table 8.10.2.3.2 Carbon Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder
Property: C Total
Method: 16

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
21	4	0.1000	0.1200	0.1075	0.0096
Means	1	0.1075	0.1075	0.1075	0.0000

Table 8.10.2.3.3 Carbon Concentration (wt. %) in Si Powder

Material: Silicon
Property: C Total
Method: 16

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
3	4	0.0580	0.0630	0.0608	0.0022
Means	1	0.0608	0.0608	0.0608	0.0000

8.10.2.4 Gravimetry

Table 8.10.2.4 Carbon Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder

Property: C Total

Method: 20

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
18	1	0.1200	0.1200	0.1200	0.0000
Means	1	0.1200	0.1200	0.1200	0.0000

8.10.3. Free Carbon in Silicon Carbide

8.10.3.1 Combustion Analysis

Table 8.10.3.1 Free Carbon Concentration (wt. %) in SiC Powder

Material: Silicon Carbide

Property: CFREE

Method: 8

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
10	3	0.6900	0.7090	0.6987	0.0096
Means	1	0.6987	0.6987	0.6987	0.0000

8.10.3.2 Electrochemical Methods - Coulometric Titration

Table 8.10.3.2 Free Carbon Concentration (wt. %) in SiC Powder

Material: Silicon Carbide

Property: CFREE

Method: 16

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
10	1	0.2700	0.2700	0.2700	0.0000
21	4	0.5700	0.6000	0.5825	0.0126
Means	2	0.2700	0.5825	0.4263	0.2210

8.10.3.3 Gravimetry

Table 8.10.3.3 Free Carbon Concentration (wt. %) in SiC Powder

Material: Silicon Carbide

Property: CFREE

Method: 20

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
22	2	0.6300	0.6600	0.6450	0.0212
Means	1	0.6450	0.6450	0.6450	0.0000

8.10.4 Carbon in Yttria-Zirconia

8.10.4.1 Combustion Analysis

Table 8.10.4.1 Carbon Concentration (wt. %) in YSZ Powder

Material: Zirconia
Property: C
Method: 8

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
15	1	0.0370	0.0370	0.0370	0.0000
17	1	0.0350	0.0350	0.0350	0.0000
22	5	0.0310	0.0390	0.0354	0.0034
Means	3	0.0350	0.0370	0.0358	0.0011

8.10.5. Nitrogen in Silicon Carbide, Silicon and Yttria-Zirconia

8.10.5.1 Combustion Analysis

Table 8.10.5.1.1 Nitrogen Concentration (wt. %) in SiC Powder

Material: Silicon Carbide
Property: N
Method: 8

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
6	3	0.0780	0.0860	0.0817	0.0040
10	1	0.1450	0.1450	0.1450	0.0000
15	1	0.0800	0.0800	0.0800	0.0000
18	1	0.0650	0.0650	0.0650	0.0000
21	3	0.0690	0.0710	0.0703	0.0012
22	2	0.0750	0.0800	0.0775	0.0035
Means	6	0.0650	0.1450	0.0866	0.0293

Table 8.10.5.1.2 Nitrogen Concentration (wt. %) in Si Powder

Material: Silicon

Property: N

Method: 8

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
3	4	0.0600	0.0700	0.0650	0.0058
6	4	0.0100	0.0200	0.0125	0.0050
15	1	0.0600	0.0600	0.0600	0.0000
18	1	0.0100	0.0100	0.0100	0.0000
Means	4	0.0100	0.0650	0.0369	0.0297

Table 8.10.5.1.3 Nitrogen Concentration (wt. %) in YSZ Powder

Material: Zirconia

Property: N

Method: 8

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
22	8	0.0220	0.0320	0.0265	0.0036
Means	1	0.0265	0.0265	0.0265	0.0000

8.10.6. Oxygen in Silicon Nitride, Silicon Carbide and Silicon

8.10.6.1 Neutron Activation Analysis

Table 8.10.6.1.1 Oxygen Concentration (wt. %) in SNR Powder

Material: Silicon Nitride, Reference Powder

Property: 0

Method: 6

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
23	12	2.0500	2.4100	2.2158	0.1252
Means	1	2.2158	2.2158	2.2158	0.0000

Table 8.10.6.1.2 Oxygen Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder

Property: 0

Method: 6

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
23	8	1.1000	1.2400	1.1613	0.0589
Means	1	1.1613	1.1613	1.1613	0.0000

Table 8.10.6.1.3 Oxygen Concentration (wt. %) in SiC Powder

Material: Silicon Carbide

Property: 0

Method: 6

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
8	2	1.3700	1.5800	1.4750	0.1485
23	8	0.7300	0.7800	0.7500	0.0169
Means	2	0.7500	1.4750	1.1125	0.5127

Table 8.10.6.1.4 Oxygen Concentration (wt. %) in Si Powder

Material: Silicon

Property: 0

Method: 6

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
8	2	1.5700	1.8200	1.6950	0.1768
23	8	0.5600	0.6900	0.6375	0.0388
Means	2	0.6375	1.6950	1.1663	0.7478

8.10.6.2 Combustion Analysis

Table 8.10.6.2.1 Oxygen Concentration (wt. %) in SNR Powder

Material: Silicon Nitride, Reference Powder (Figure 74)

Property: 0

Method: 8

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	6	2.4490	2.5680	2.5300	0.0488
3	8	2.3100	2.3300	2.3175	0.0089
5	1	1.9000	1.9000	1.9000	0.0000
6	4	2.0400	2.2000	2.1325	0.0727
7	6	2.1900	2.3200	2.2650	0.0472
13	11	2.1100	2.2800	2.1745	0.0573
14	1	2.0000	2.0000	2.0000	0.0000
15	1	2.4000	2.4000	2.4000	0.0000
16	4	1.7500	2.0400	1.9000	0.1463
17	2	2.2100	2.2300	2.2200	0.0141
18	2	2.0500	2.1300	2.0900	0.0566
19	1	2.0900	2.0900	2.0900	0.0000
21	6	2.1800	2.3700	2.2533	0.0647
22	12	2.1300	2.4200	2.2525	0.0984
24	2	2.0800	2.1000	2.0900	0.0141
25	3	2.5100	2.6900	2.6000	0.0900
Means	16	1.9000	2.6000	2.2010	0.1991

Table 8.10.6.2.2 Oxygen Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder (Figure 75)

Property: 0

Method: 8

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	4	1.5500	2.5900	2.0675	0.5976
5	4	1.0800	1.1900	1.1300	0.0455
6	2	1.3300	1.4800	1.4050	0.1061
7	3	1.3400	1.3600	1.3533	0.0115
14	1	1.1000	1.1000	1.1000	0.0000
15	1	1.2400	1.2400	1.2400	0.0000
16	12	1.1070	1.2530	1.2068	0.0434
18	2	1.2000	1.2500	1.2250	0.0354
19	7	1.1900	1.2600	1.2271	0.0206
21	6	1.1600	1.2100	1.1783	0.0172
22	4	1.2500	1.2800	1.2625	0.0150
24	14	1.0400	1.1800	1.1150	0.0372
25	5	1.3030	1.3780	1.3410	0.0275
Means	13	1.1000	2.0675	1.2963	0.2495

Table 8.10.6.2.3 Oxygen Concentration (wt. %) in SiC Powder

Material: Silicon Carbide (Figure 76)

Property: 0

Method: 8

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
2	2	0.6750	0.6750	0.6750	0.0000
6	3	0.7330	0.7490	0.7397	0.0083
7	2	0.7300	0.7400	0.7350	0.0071
10	1	0.8250	0.8250	0.8250	0.0000
15	1	0.4500	0.4500	0.4500	0.0000
21	3	0.6900	0.7100	0.7000	0.0100
22	2	0.6400	0.6450	0.6425	0.0035
25	13	0.7160	0.7650	0.7387	0.0145
Means	8	0.4500	0.8250	0.6882	0.1104

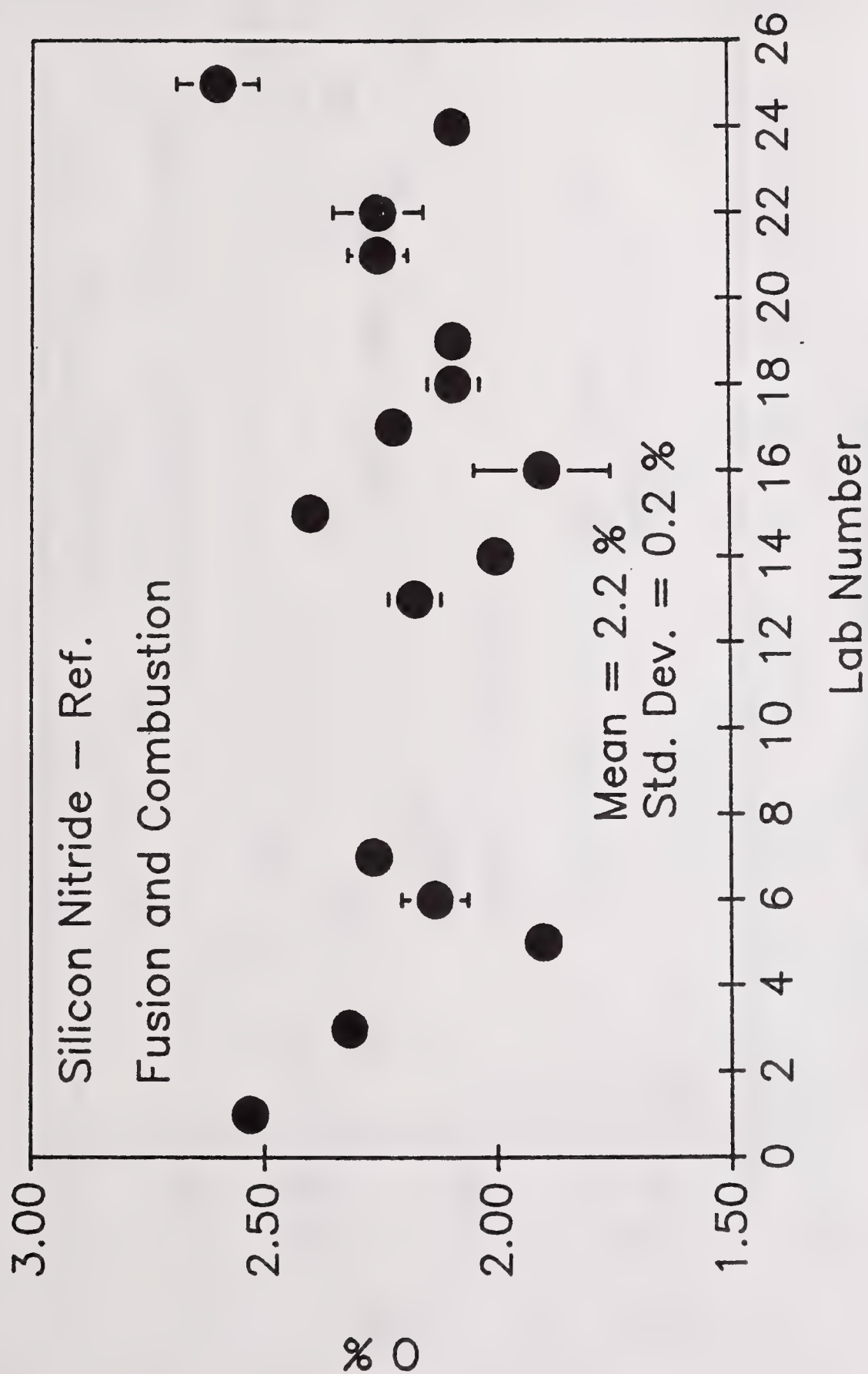


Figure 74. Oxygen concentration (wt. %) in SNR powder as reported by labs using Combustion Analysis

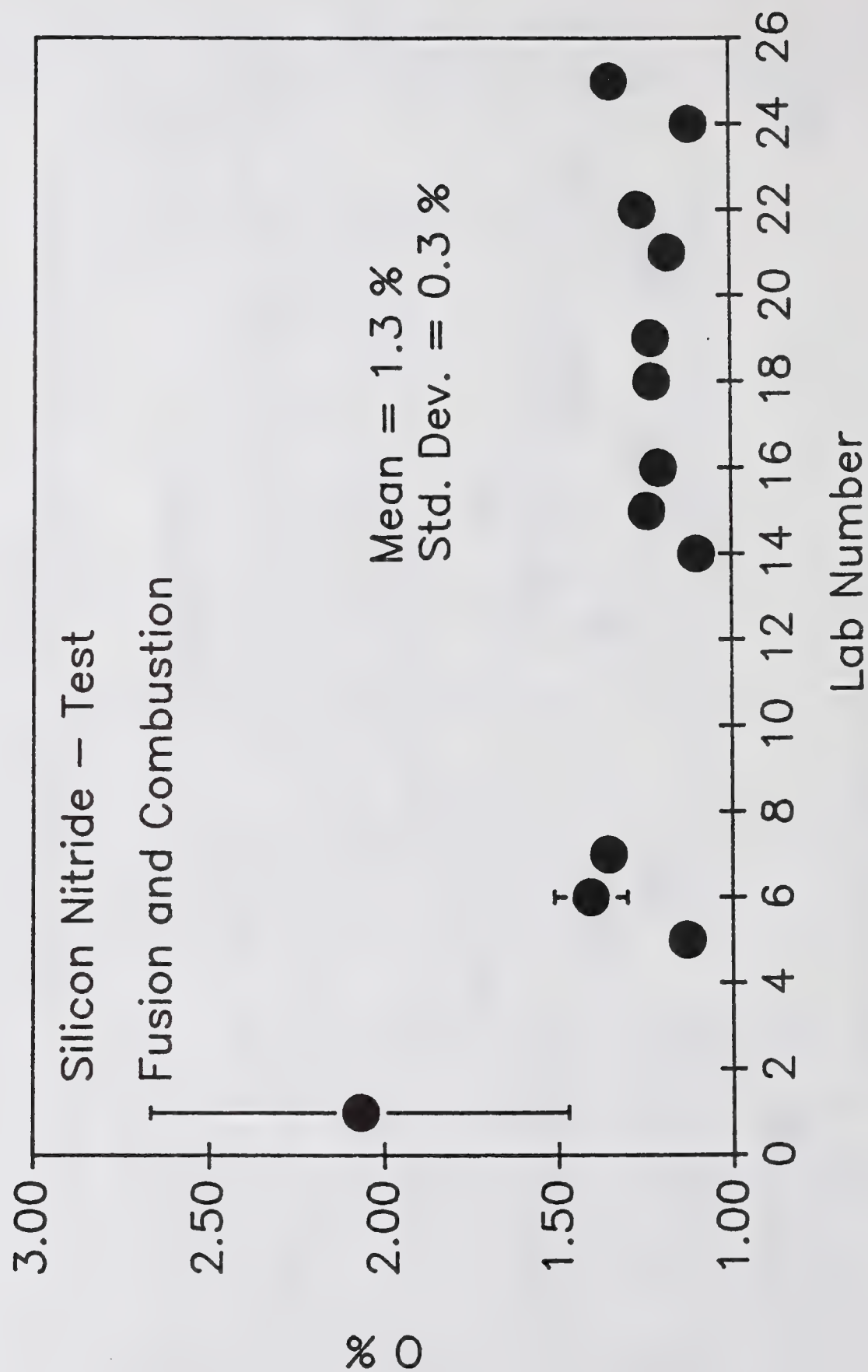


Figure 75. Oxygen concentration (wt. %) in SNT powder as reported by labs using Combustion Analysis

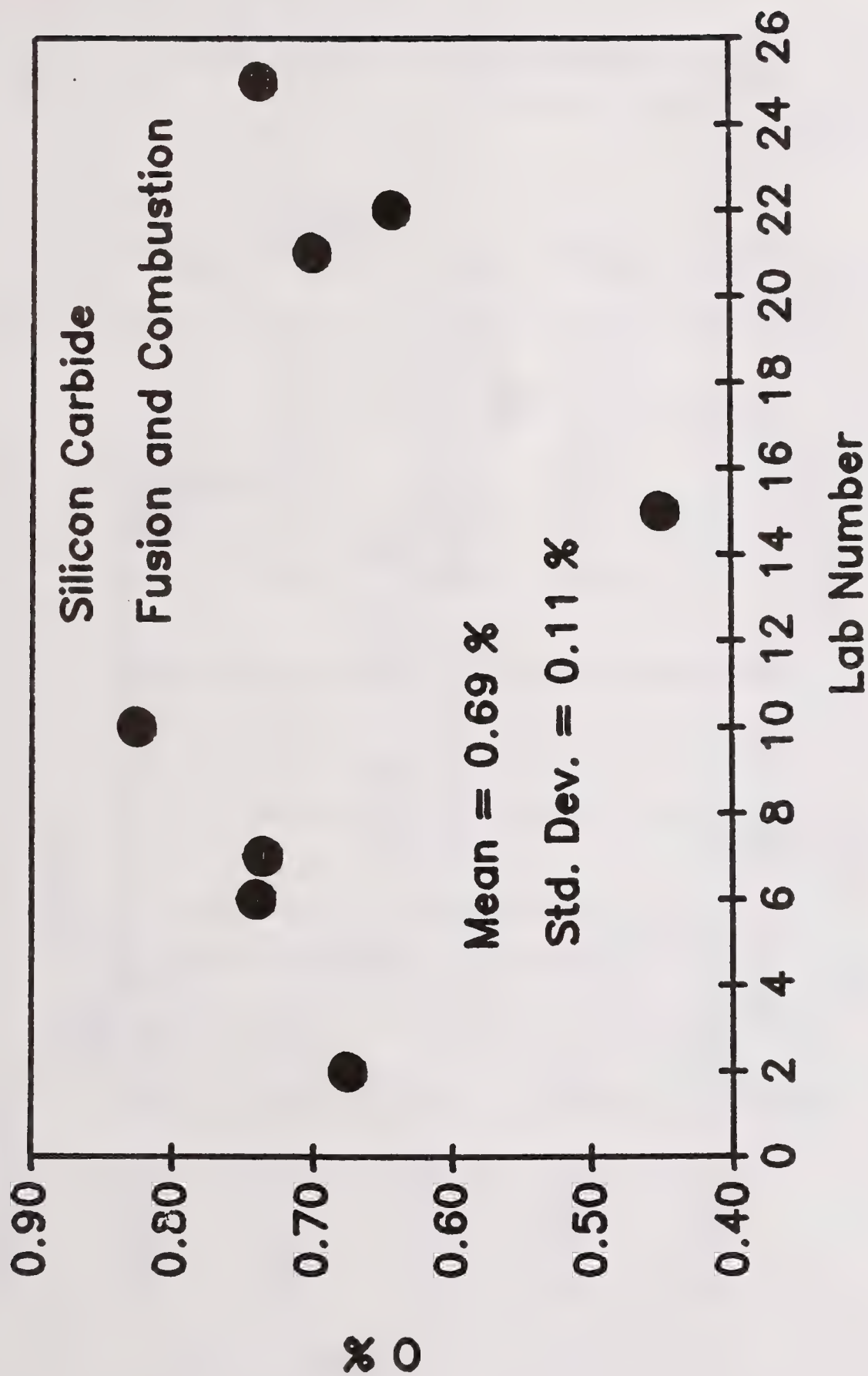


Figure 76. Oxygen concentration (wt. %) in SiC powder as reported by labs using Combustion Analysis

Table 8.10.6.2.4 Oxygen Concentration (wt. %) in Si Powder

Material: Silicon (Figure 77)

Property: 0

Method: 8

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
2	2	0.5100	0.5100	0.5100	0.0000
3	4	0.5400	0.5560	0.5478	0.0075
5	4	0.4900	0.5500	0.5075	0.0287
6	4	0.4700	0.5500	0.4975	0.0359
15	1	0.5400	0.5400	0.5400	0.0000
16	5	0.5160	0.5490	0.5352	0.0138
18	1	0.4900	0.4900	0.4900	0.0000
22	4	0.5100	0.5200	0.5175	0.0050
Means	8	0.4900	0.5478	0.5182	0.0209

8.10.6.3 Electrochemical Methods - Coulometric Titration

Table 8.10.6.3 Oxygen Concentration (wt. %) in SNR Powder

Material: Silicon Nitride, Reference Powder

Property: 0

Method: 16

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
2	3	1.7800	1.9800	1.8967	0.1041
Means	1	1.8967	1.8967	1.8967	0.0000

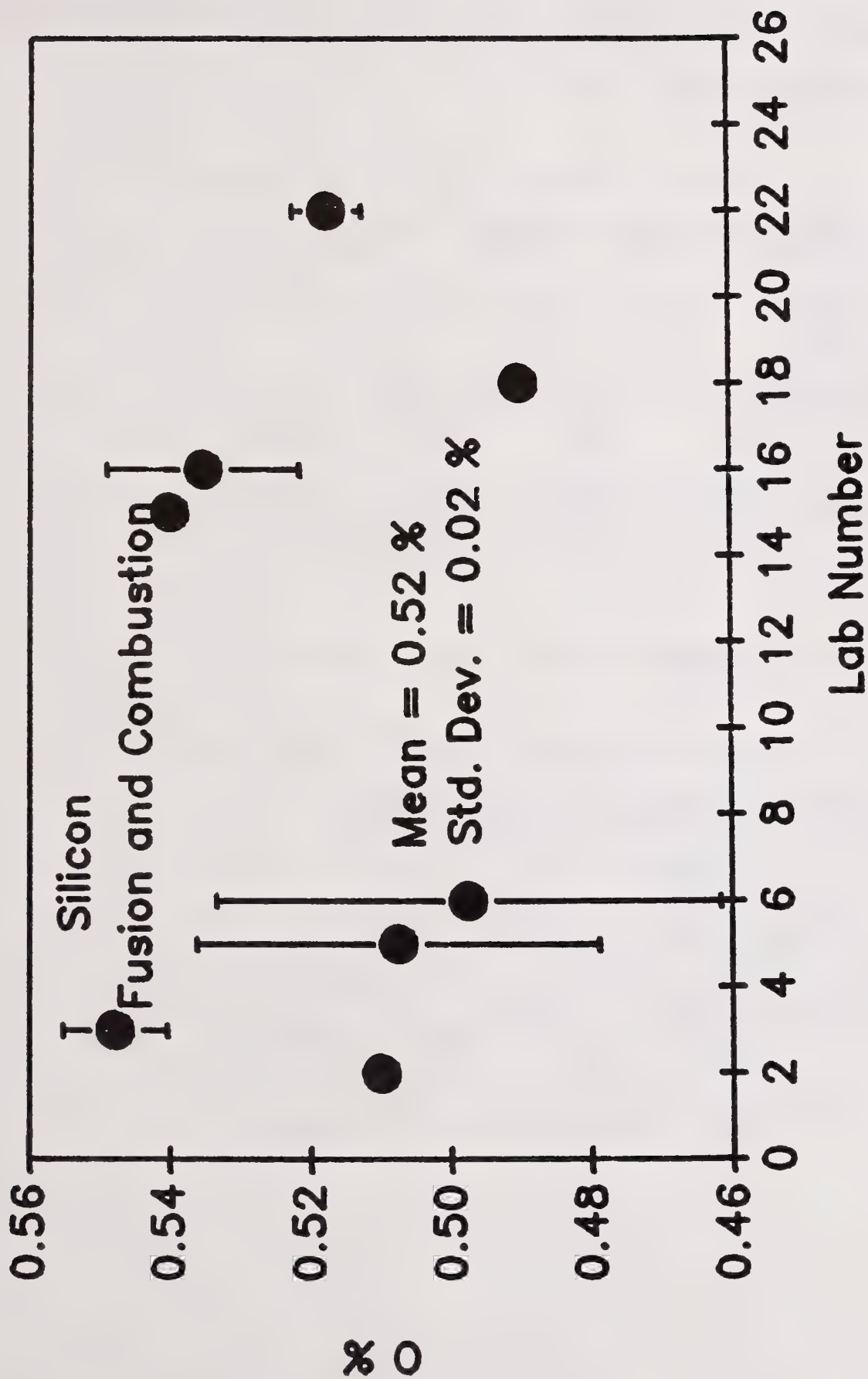


Figure 77. Oxygen concentration (wt. %) in Si powder as reported by labs using Combustion Analysis

8.10.7. Fluorine in Silicon Nitride and Silicon Carbide

8.10.7.1 Mass Spectrometry

Table 8.10.7.1 Fluorine Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder

Property: F

Method: 5

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
8	3	0.0000	0.0000	0.0000	0.0000
Means	1	0.0000	0.0000	0.0000	0.0000

8.10.7.2 Ion Chromatography

Table 8.10.7.2.1 Fluorine Concentration (wt. %) in SNR Powder

Material: Silicon Nitride, Reference Powder

Property: F

Method: 3

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
24	3	0.1100	0.1500	0.1293	0.0200
25	1	0.1700	0.1700	0.1700	0.0000
Means	2	0.1293	0.1700	0.1497	0.0288

Table 8.10.7.2.2 Fluorine Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder

Property: F

Method: 3

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
24	4	0.0020	0.0020	0.0020	0.0000
25	6	0.0070	0.0080	0.0075	0.0005
Means	2	0.0020	0.0075	0.0047	0.0039

8.10.7.3 Electrochemical Methods - Potentiometric Titration

Table 8.10.7.3.1 Fluorine Concentration (wt. %) in SNR Powder

Material: Silicon Nitride, Reference Powder

Property: F

Method: 13

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
5	1	0.1200	0.1200	0.1200	0.0000
14	2	0.1200	0.1200	0.1200	0.0000
21	2	0.0760	0.0800	0.0780	0.0028
Means	3	0.0780	0.1200	0.1060	0.0242

Table 8.10.7.3.2 Fluorine Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder

Property: F

Method: 13

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
5	4	0.0040	0.0040	0.0040	0.0000
14	2	0.0060	0.0060	0.0060	0.0000
21	2	0.0050	0.0050	0.0050	0.0000
Means	3	0.0040	0.0060	0.0050	0.0010

Table 8.10.7.3.3 Fluorine Concentration (wt. %) in SiC Powder

Material: Silicon Carbide

Property: F

Method: 13

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
21	3	0.0400	0.0420	0.0407	0.0012
Means	1	0.0407	0.0407	0.0407	0.0000

8.10.7.4 Electrochemical Methods - Specific Ion Electrode

Table 8.10.7.4.1 Fluorine Concentration (wt. %) in SNR Powder

Material: Silicon Nitride, Reference Powder

Property: F

Method: 4

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
6	2	0.1500	0.1640	0.1570	0.0099
Means	1	0.1570	0.1570	0.1570	0.0000

Table 8.10.7.4.2 Fluorine Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder

Property: F

Method: 4

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
6	1	0.0110	0.0110	0.0110	0.0000
Means	1	0.0110	0.0110	0.0110	0.0000

Table 8.10.7.4.3 Fluorine Concentration (wt. %) in SiC Powder

Material: Silicon Carbide

Property: F

Method: 4

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
6	1	0.0730	0.0730	0.0730	0.0000
Means	1	0.0730	0.0730	0.0730	0.0000

8.10.8 Sulfur in Yttria-Zirconia8.10.8.1 Mass Spectrometry

Table 8.10.8.1 Sulfur Concentration (wt. %) in YSZ Powder

Material: Zirconia

Property: S

Method: 5

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
8	3	0.2000	0.2000	0.2000	0.0000
Means	1	0.2000	0.2000	0.2000	0.0000

8.10.8.2 Combustion Analysis

Table 8.10.8.2 Sulfur Concentration (wt. %) in YSZ Powder

Material: Zirconia
Property: S
Method: 8

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
14	2	0.0900	0.0900	0.0900	0.0000
15	1	0.0910	0.0910	0.0910	0.0000
17	1	0.1000	0.1000	0.1000	0.0000
Means	3	0.0900	0.1000	0.0937	0.0055

8.10.8.3 Inductively Coupled Plasma - Emission Spectroscopy

Table 8.10.8.3 Sulfur Concentration (wt. %) in YSZ Powder

Material: Zirconia
Property: S
Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
6	1	0.1160	0.1160	0.1160	0.0000
Means	1	0.1160	0.1160	0.1160	0.0000

8.10.8.4 Ion Chromatography

Table 8.10.8.4 Sulfur Concentration (wt. %) in YSZ Powder

Material: Zirconia

Property: S

Method: 3

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
6	1	0.0310	0.0310	0.0310	0.0000
24	3	0.0160	0.0210	0.0180	0.0026
Means	2	0.0180	0.0310	0.0245	0.0092

8.10.9 Chlorine in Yttria-Zirconia

8.10.9.1 Neutron Activation Analysis

Table 8.10.9.1 Chlorine Concentration (wt. %) in YSZ Powder

Material: Zirconia

Property: Cl

Method: 6

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
2	1	0.0340	0.0340	0.0340	0.0000
Means	1	0.0340	0.0340	0.0340	0.0000

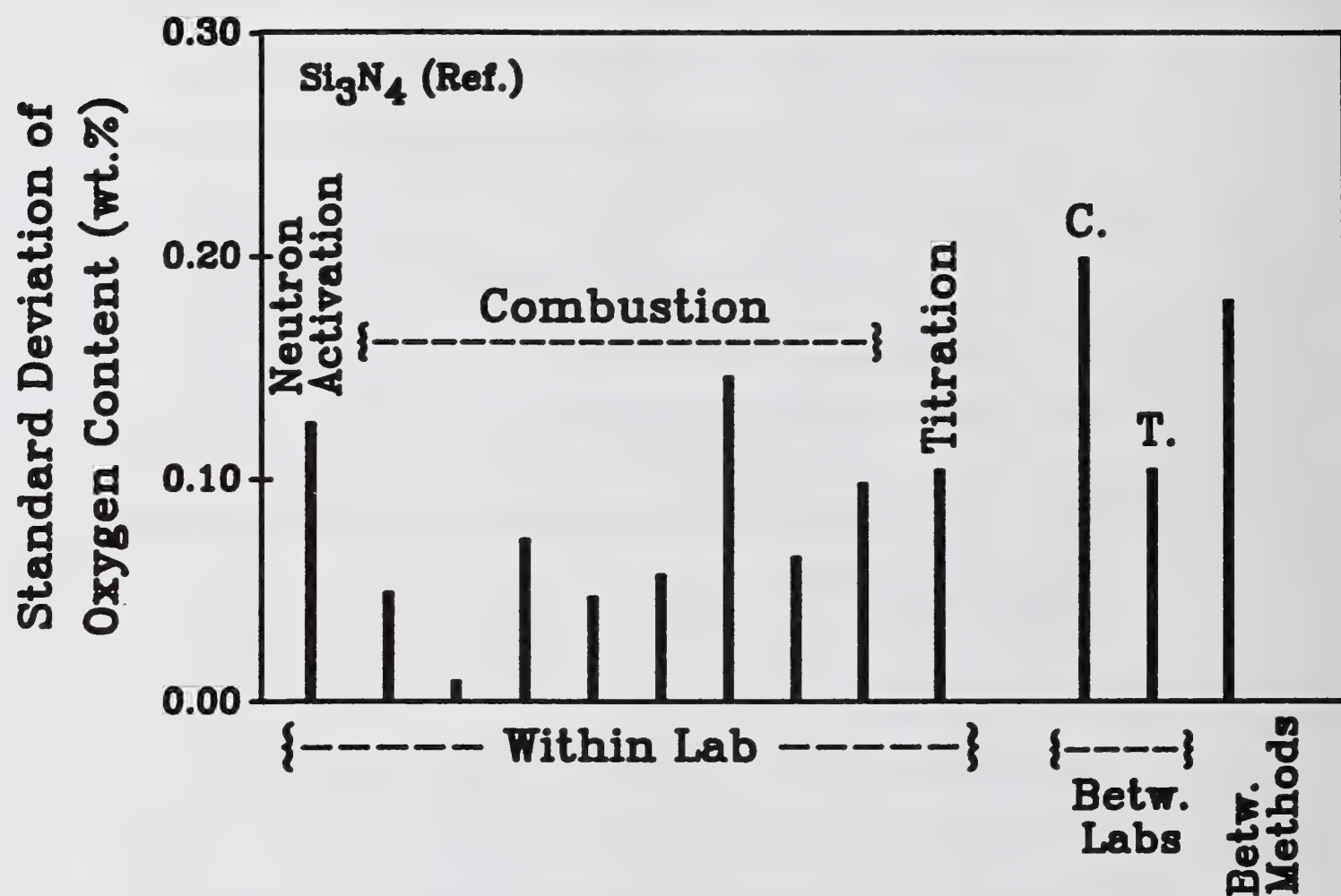


Figure 78. Within lab, between lab, and between methods standard deviations in the measurement of oxygen content for the Si₃N₄(Ref.) powder as measured by various laboratories using a variety of methods.

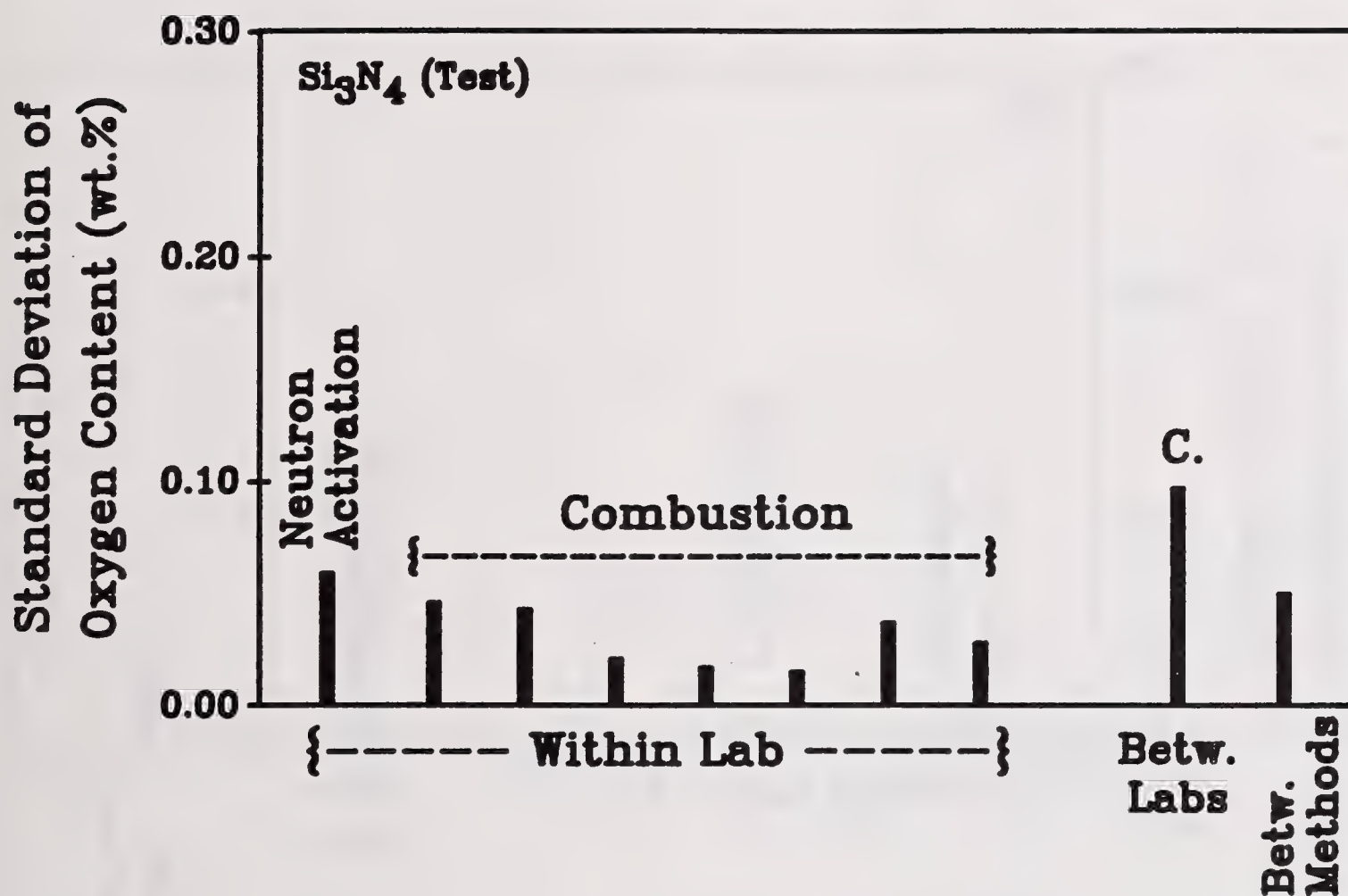


Figure 79. Within lab, between lab, and between methods standard deviations in the measurement of oxygen content for the Si₃N₄(Test) powder as measured by various laboratories using a variety of methods.

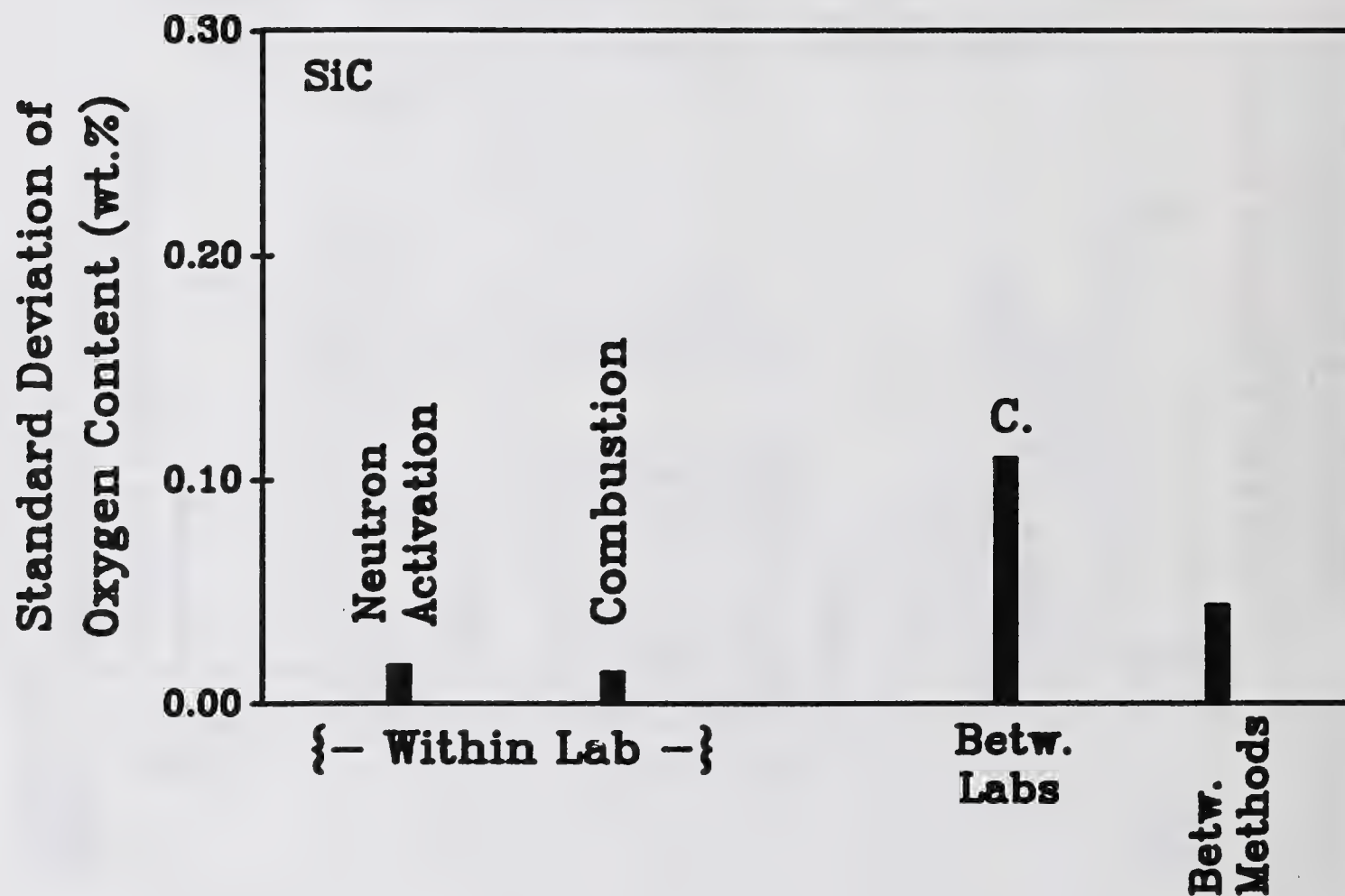


Figure 80. Within lab, between lab, and between methods standard deviations in the measurement of oxygen content for the SiC powder as measured by various laboratories using a variety of methods.

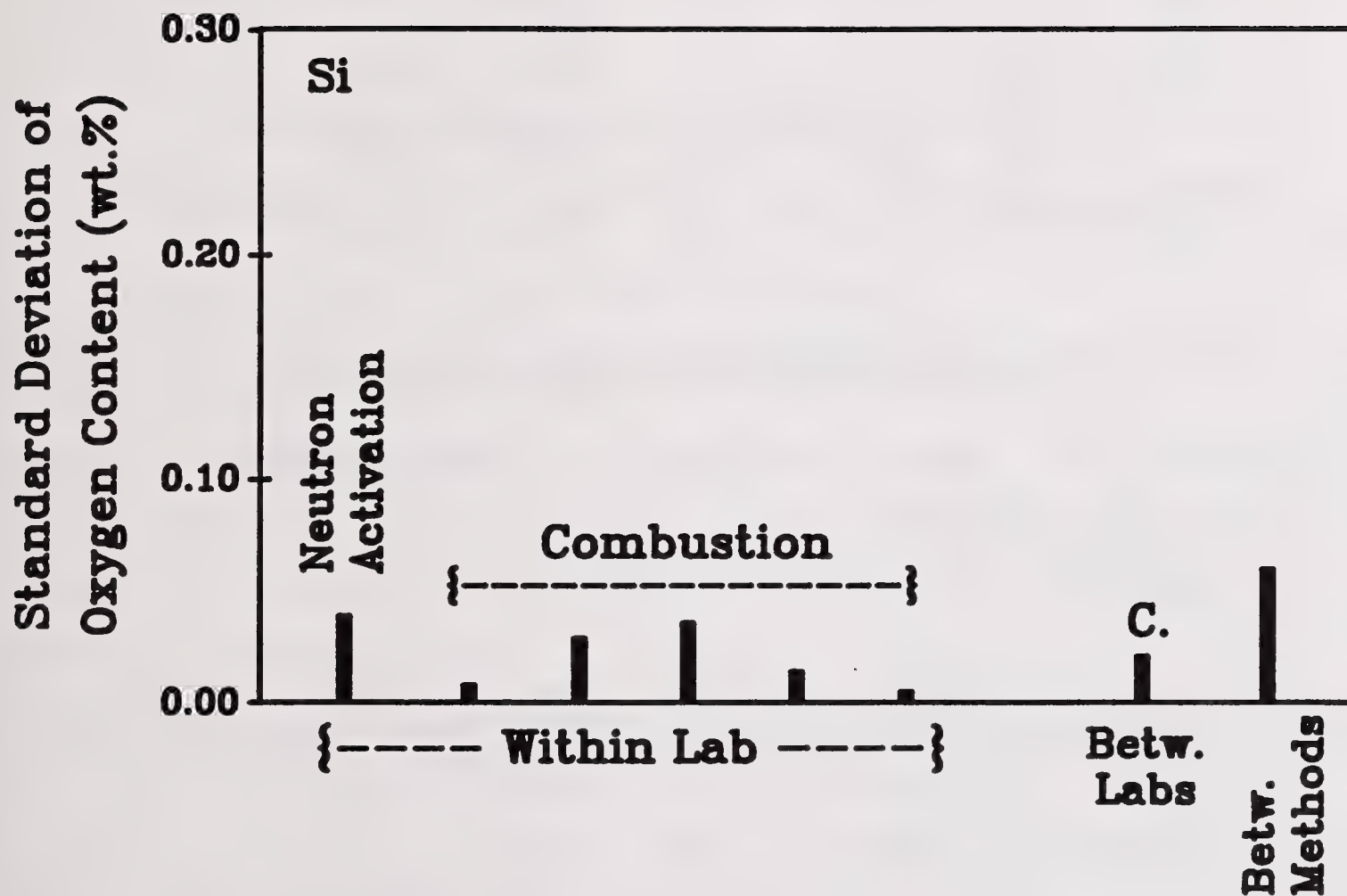


Figure 81. Within lab, between lab, and between methods standard deviations in the measurement of oxygen content for the Si powder as measured by various laboratories using a variety of methods.

8.10.9.2 Ion Chromatography

Table 8.10.9.2 Chlorine Concentration (wt. %) in YSZ Powder

Material: Zirconia

Property: Cl

Method: 3

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
6	2	0.0180	0.8600	0.4390	0.5954
24	3	0.0250	0.0280	0.0260	0.0017
Means	2	0.0260	0.4390	0.2325	0.2920

8.10.9.3 Electrochemical Methods - Argentometric Titration

Table 8.10.9.3 Chlorine Concentration (wt. %) in YSZ Powder

Material: Zirconia

Property: Cl

Method: 14

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
14	1	0.0220	0.0220	0.0220	0.0000
Means	1	0.0220	0.0220	0.0220	0.0000

8.11. STATISTICAL ANALYSIS, R. Munro, NIST

The major nonmetallic impurities in the powders were carbon, nitrogen, and oxygen, whenever they were not major constituents, along with chlorine, fluorine, and sulfur. Two of the methods used for detecting these elements, combustion and coulometric titration, involved the decomposition of the powders in chemical reactions. A third method, fast neutron activation analysis, provided an alternative technique that did not require a decomposition reaction. Use of the latter method, however, was very limited because of the need for a nuclear reactor facility, and there were insufficient results to obtain a valid measure of its reproducibility. Results for oxygen as an impurity, Figures 78-81, indicated that all the methods were able to achieve approximately the same level of precision.

9. MAJOR METALLIC IMPURITIES

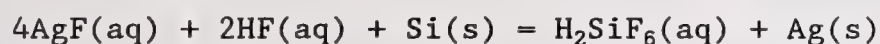
A. L. Dragoo, S. G. Malghan, R. G. Munro and S. M. Hsu; NIST

9.1. METHODS FOR FREE SILICON IN SILICON NITRIDE

Three determinations of free silicon in silicon nitride - 2 samples of SNT powder and 1 of SNR powder - were reported. Each determination was carried out by a different method. Although the data were insufficient for analysis, a brief summary of the methods is of interest. Data are summarized in Section 8.10.1.

9.1.1. Electrochemical Methods - Potentiometric Titration (See 7.2.3.)

This method was reported by Lab 16. The silicon nitride sample was leached with an AgF/HF solution. Free Si dissolves according to the reaction



The free silver was dissolved in nitric acid, and the amount of AgNO_3 determined by potentiometric titration with a standardized KCl solution.

9.1.2. Volumetric Analysis

Lab 21 reported the use of this method. A silicon nitride powder sample was reacted with a solution of NaOH. H_2 formed by the reaction of NaOH with free Si was collected and the volume of gas evolved was measured.

9.1.3. Standardized Method

A method described in DIN 51075, Part 2, for the determination of free Si in SiC was used by Lab 3 for the analysis of free Si in the SNR powder.

Conclusions

Data were insufficient for adequate comparison of the methods.

9.2. METHODS FOR ALUMINUM, CALCIUM AND IRON

Aluminum and iron were determined as major impurities in SNR, SiC, Si and YSZ powders and possibly as a major impurity in the SNT powder. Ca was determined as a minor impurity in the Si powder.

ICP was the primary measurement method used, but AAS and DCP also were used significantly for the analysis of the SNR powder. One or two measurements were reported by XRF, optical emission spectroscopy and mass spectrometry. However, the data obtained by these latter methods were insufficient for statistical evaluation.

Data are summarized in Section 9.4.1. for Al, in Section 9.4.2. for Ca, and in Section 9.4.3. for Fe.

9.2.1. Atomic Absorption Spectroscopy (See 7.1.2.)

Instrument and Experimental Parameters

The predominant method of sample preparation for Si_3N_4 was acid digestion in a pressure bomb. HF-HNO_3 was the usual acid mixture; one laboratory reported the addition of HClO_4 as a third component. The sample was reacted at 160 to 180 °C for 8 to 16 h. The solution which was obtained was usually treated with HClO_4 and fumed to dryness to remove Si as SiF_4 . The residue was dissolved, for example, in 5% hydrogen iodide and diluted to a standard volume.

Flux decomposition was used by a smaller number of laboratories for the dissolution of Si_3N_4 . $\text{NaO}_2\text{-Na}_2\text{CO}_3$, KOH and $\text{Na}_2\text{B}_4\text{O}_7$ were used as fluxes. The glass bead was dissolved in H_2SO_4 and the solution treated with HClO_4 . The solution was fumed to precipitate SiO_2 , which was filtered off. The volume and pH of the solution were adjusted.

SiC also was decomposed primarily with acids. A pressure bomb apparently was not used by all laboratories. One laboratory reported using a $\text{HF-HNO}_3\text{-H}_2\text{SO}_4$ (1:3:5) mixture and conducting the reaction at 210 °C for 12 h. Flux decomposition using Li_2CO_3 was reported by one laboratory.

One analyst noted that often the procedures for SiC and Si_3N_4 are the same; however, many times the SiC did not dissolve in the pressure bomb. When this happened, $\text{NaO}_2\text{-Na}_2\text{CO}_3$ was used. Even this was not a completely satisfactory method.

In preparing Si samples for AAS, ICP or DCP analysis, most laboratories decomposed the Si samples by dissolution in an acid solution which usually consisted of a mixture of HF and HNO_3 or HF and HCl . A pressure bomb was used in some cases. After dissolution, H_2SO_4 or HClO_4 was added, and the solution was fumed to remove Si.

With regard to preparation of Si samples by the flux method, one laboratory reported decomposing samples in a $\text{NaO}_2\text{-Na}_2\text{CO}_3$ flux; another laboratory reported the use of a $\text{Na}_2\text{P}_4\text{O}_7$ flux.

Results and Discussion

A comparison of the sample dissolution and detection methods for the determination of Al, Ca and Fe is presented in Table 9.2.1 for the SNR powder, in Table 9.2.2 for the SNT powder and in Table 9.2.3 for the SiC powder. Results for the determination of Al and Fe in Si powder are presented in Table 9.2.4.

In general AAS, ICP and DCP yielded very consistent results for Si_3N_4 when acid digestion in a pressure bomb was used for the preparation of silicon nitride and silicon carbide samples.

AAS and ICP gave consistent results when acid decomposition was used for the preparation of Si samples.

Conclusions

See Section 7.1.2.

9.2.2. Inductively Coupled Plasma (See 7.1.3.)

Instrument and Experimental Parameters

Laboratory §8 reported the following operating conditions:

Coolant gas	12 L/min
Plasma gas	0.8 L/min
Nebulizer gas	0.8 L/min
Sample uptake	1.1 mL/min
Power	1200 W
Frequency	17.2 MHz

This laboratory also reported the addition of 10 $\mu\text{g/g}$ Sc as an internal standard and the use of synthetic standard solutions for calibration.

Results and Discussion

A comparison of the sample dissolution and detection methods for the two silicon nitride powders, silicon carbide and silicon is presented in Tables 9.2.2.1, 9.2.2.2, 9.2.2.3 and 9.2.2.4, respectively.

In general AAS, ICP and DCP yielded very consistent results for Si_3N_4 when acid digestion in a pressure bomb was used.

Conclusions

Acid decomposition as described in Section 7.1.3 gave the most consistent results. Results obtained with flux decomposition were frequently high with respect to the results from acid decomposition.

AAS, ICP and DCP gave comparable results. The best precision is achieved when an internal standard is incorporated into the solution and when synthetic standard solutions are used for calibration.

9.2.3. Optical Emission Spectroscopy (OES)

Use and Significance

Since classical spectrochemical analysis was usually conducted with an arc emission spectrograph, analysis by this method is often termed "spectrochemical analysis." However, today spectrochemical analysis may be carried out by other methods, such as AAS, ICP and DCP. To avoid confusion, "spectrochemical analysis" or "spectrochemical methods" will only be used in the generic sense in this report.

For qualitative analysis, the emission spectrograph is capable of detecting 0.001% or less of most metallic elements and certain nonmetallic ones, including P, Si, As, C and B, in a few milligrams of material. Quantitative analysis can be conducted with automatic recording spectrographs. The typical concentration range is normally 0.0001% to 75%; however, quantitative precision is badly degraded by concentrations above the 30% level¹⁰.

Instrument and Experimental Parameters

The steps in emission spectroscopy are sample excitation, radiation dispersion, radiation detection, data recording, data processing and information reporting. The arc is powered and controlled by a high-voltage arc generator. The control parameters are the self-inductance, L , of the discharge circuit; and the resistance, R , of the discharge circuit; the capacitance, C , of the charging capacitor; the voltage, V_c , at the capacitor; and the voltage, V_g , at the gap. By adjusting combinations of V_c , V_g , C , L and R , the energy consumed by the spark and the duration of the discharge, reproducible spectrochemical characteristics can be generated.

Since emission spectrographic methods are relative rather than absolute, calibration with high-quality standards is required. Alteration of conditions which affect the consistency of results necessitates the recalibration of the instrument. For a high level of accuracy for a method, all samples should exhibit similar rates of volatilization and spectral emission lines. Standards and samples must be matched in physical and chemical composition. Differences occurring during excitation must be minimized by the use of an internal standard.

For optimum results, analysis of refractories, sinters and ores requires a short time duration for the spark.

Details were not reported for preparative methods used in this study.

Results and Discussion

The single determination of Al in SNR Si_3N_4 is high with respect to results given by other methods. The result reported for Fe in SNR Si_3N_4 also may be slightly high with respect to the other methods, but the limited amount of data does not permit a valid comparison to be made. The result reported for SNT Si_3N_4 appears to be in agreement with results obtained by NAA and DCP, but again the limited amount of data does not permit valid comparisons to be made. The result reported for Al in SiC appears to be low with respect to other spectroscopic methods; whereas, the value for Ca is difficult to compare due to the large amount of uncertainty in the results by all methods. Again, the limited data make any comparison subject to question.

Conclusions

Data were insufficient for an analysis of variance and for valid comparisons to be made with other data.

Table 9.2.2.1 Effects of Sample Dissolution Method and Spectrochemical Analysis Method Measured Concentrations of Al, Ca and Fe in SNR Si_3N_4 . Concentrations are in percent by weight. Uncertainties in parentheses are given as Student's t (95% confidence level) to adjust for the different numbers of determinations reported.

METHOD	ACID	FLUX
<u>Al in SNR Si_3N_4</u>		
AAS	0.040 (0.013)	0.030 (--)
ICP	0.040 (0.016)	0.037 (0.026)
DCP	0.042 (0.013)	0.040 (--)
<u>Ca in SNR Si_3N_4</u>		
AAS	0.011 (0.014)	0.06 (0.10)
ICP	0.010 (0.014)	0.010 (--)
DCP	0.009 (0.011)	0.030 (--)
<u>Fe in SNR Si_3N_4</u>		
AAS	0.020 (0.010)	0.026 (0.035)
ICP	0.019 (0.014)	0.02 (0.09)
DCP	0.023 (0.013)	0.040 (--)

Table 9.2.2.2 Effects of Sample Dissolution Method and Spectrochemical Analysis Method Measured Concentrations of Al, Ca and Fe in SNT Si₃N₄. Concentrations are in percent by weight. Uncertainties in parentheses are given as Student's t (95% confidence level) to adjust for the different numbers of determinations reported.

METHOD	ACID	FLUX
<u>Al in SNT Si₃N₄</u>		
AAS	0.0011 (0.0013)	NA
ICP	0.0003 (0.0013)	NA
DCP	0.0016 (0.0013)	NA
<u>Ca in SNT Si₃N₄</u>		
AAS	0.0032 (--)	0.04 (0.14)
ICP	0.0006 (0.0011)	NA
DCP	0.0029 (0.0013)	NA
<u>Fe in SNT Si₃N₄</u>		
AAS	0.0049 (0.0043)	0.033 (0.013)
ICP	0.0040 (0.0013)	NA
DCP	0.0040 (0.0014)	NA

NA, not available

Table 9.2.2.3 Effects of Sample Dissolution Method and Spectrochemical Analysis Method Measured Concentrations of Al, Ca and Fe in SiC. Concentrations are in percent by weight. Uncertainties in parentheses are given as Student's t (95% confidence level) to adjust for the different numbers of determinations reported.

METHOD	ACID	FLUX
<u>Al in SiC</u>		
AAS	0.025 (0.013)	NA
ICP	0.048 (0.040)	0.06 (0.7)
DCP	0.044 (0.005)	NA
<u>Ca in SiC</u>		
AAS	0.009 (0.02)	NA
ICP	0.011 (0.020)	0.13 (0.19)
DCP	0.008 (0.002)	NA
<u>Fe in SiC</u>		
AAS	0.0015 (0.0014)	NA
ICP	0.007 (0.010)	0.1 (1.2)
DCP	0.0027 (0.0016)	NA
NA, not available		

Table 9.2.2.4 Effects of Sample Dissolution Method and Spectrochemical Analysis Method on Measured Concentrations of Al and Fe in Si. Concentrations are in percent by weight. Uncertainties in parentheses are estimated as Student's t (95 % confidence level) to adjust for the different number of determinations reported.

METHOD	ACID	FLUX
<u>Al in Si</u>		
AAS	0.024 (0.005)	0.030 (--)
ICP	0.026 (0.021)	NA
DCP	NA	0.043 (0.003)
<u>Fe in Si</u>		
AAS	0.042 (0.006)	0.050 (--)
ICP	0.040 (0.013)	0.1 (1.2)
DCP	NA	0.050 (0.029)
NA, not available		

9.2.4. DC Plasma (See 7.1.4.)

Instrument and Experimental Parameters

With regard to sample preparation, see Section 9.2.1.

Czupryna and Natansohn¹¹ discussed experimental conditions for the analysis of major metallic impurities in Si_3N_4 . They employed an alkali fusion technique to decompose their samples: namely, $\text{Na}_2\text{CO}_3 + \text{Na}_2\text{SO}_4$. The fused material was dissolved in 1 M nitric acid. They observed that the DCP measurement is affected by the following factors:

- Total dissolved solids content which affects the amount of background signal and broadening of emission lines;
- Spectral line overlap;
- Physico-chemical (matrix) effects which affect the ionization of the system and either enhance or suppress the analyte signal.

In particular they investigated the suppression of Ca and Mg signals by Si. They determined the detectability limits shown in Table 9.2.4.1.

Results and Discussion

See Tables 9.2.2.1 to 4.

Flux decomposition was the only sample preparation method used to prepare Si samples for DCP analysis. Results were high for both Ca and Fe determinations as compared to results obtained with either AAS or ICP and acid decomposition. This bias is suspected to be due to the flux decomposition method.

Conclusion

See Section 9.2.2.

Table 9.2.4.1 Detectability for Impurity Measurements in Si_3N_4 .
Measurements made in 24 g/L of alkaline flux; Si_3N_4 at 1 mg/mL
(from Czupryna and Natansohn, Ref. 1)

Element	Wavelength (nm)	Measured in Solution ($\mu\text{g/mL}$)	Determinable in Solid ($\mu\text{g/g}$)
Al	396.15	0.01	10
B	249.77	0.10	100
Ca	393.37	0.010	10
Cr	425.43	0.001	1
Fe	371.99	0.10	100
Mg	279.55	0.01	10
Mo	379.82	0.010	10
W	400.87	0.10	100

9.3. METHODS FOR OTHER MAJOR METALLIC IMPURITIES IN YTTRIA-ZIRCONIA (Si, Ti, Hf, Sn, Nd, Ho)

The results obtained by the various methods are summarized in Table 9.3.1. Si and Hf were the only impurities measured by at least 3 laboratories. In both cases, ICP was the method used. For most of the results only one determination per lab was reported. These determinations are not evaluated here due to insufficient.

Table 9.3.1

Major Impurities in Yttria-Zirconia, % Weight

Method	Si	Ti	Hf	Sn	Nd	Ho
NAA	NA	0.026	NA	NA	NA	NA
MS	NA	NA	0.60	NA	NA	NA
XRF	NA	0.025	1.83	NA	NA	NA
AAS	0.016	0.046	1.86	0.04	0.028	0.025
ICP	0.007	0.024	1.32	NA	NA	NA
DCP	0.01	0.025	1.76	NA	NA	NA

NA, not available

9.4. STATISTICAL DATA ON MAJOR METALLIC IMPURITIES9.4.1 Aluminum in All Powders9.4.1.1 Mass Spectrometry

Table 9.4.1.1.1 Aluminum Concentration (wt. %) in SiC Powder

Material: Silicon Carbide

Property: Al

Method: 5

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
8	2	0.0040	0.0050	0.0045	0.0007
Means	1	0.0045	0.0045	0.0045	0.0000

Table 9.4.1.1.2 Aluminum Concentration (wt. %) in Si Powder

Material: Silicon

Property: Al

Method: 5

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
8	2	0.015	0.015	0.015	0.000
Means	1	0.015	0.015	0.015	0.000

Table 9.4.1.1.3 Aluminum Concentration (wt. %) in ZrO₂ Powder

Material: Zirconia

Property: Al

Method: 5

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
8	3	0.003	0.050	0.023	0.024
Means	1	0.023	0.023	0.023	0.000

9.4.1 Aluminum in All Powder9.4.1.2 Neutron Activation Analysis

Table 9.4.1.2 Aluminum Concentration (wt. %) in YSZ Powder

Material: Zirconia

Property: Al

Method: 6

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
2	1	0.046	0.046	0.046	0.000
Means	1	0.046	0.046	0.046	0.000

9.4.1.3 X-ray Fluorescence Spectrometry

Table 9.4.1.3.1 Aluminum Concentration (wt. %) in SNR Powder

Material: Silicon Nitride, Reference Powder
Property: Al
Method: 12

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
5	1	0.038	0.038	0.038	0.000
Means	1	0.038	0.038	0.038	0.000

Table 9.4.1.3.2 Aluminum Concentration (wt. %) in SiC Powder

Material: Silicon Carbide
Property: Al
Method: 12

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
10	1	0.026	0.026	0.026	0.000
Means	1	0.026	0.026	0.026	0.000

Table 9.4.1.3.3 Aluminum Concentration (wt. %) in YSZ Powder

Material: Zirconia
Property: Al
Method: 12

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
24	3	0.042	0.042	0.042	0.000
Means	1	0.042	0.042	0.042	0.000

9.4.1. Aluminum in All Powder

9.4.1.4 Atomic Absorption Spectroscopy

Table 9.4.1.4.1 Aluminum Concentration (wt. %) in SNR Powder

Material: Silicon Nitride, Reference Powder (Figure 82)
 Property: Al
 Method: 1

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	1	0.030	0.030	0.030	0.000
14	2	0.044	0.045	0.044	0.000
16	6	0.035	0.042	0.038	0.002
18	1	0.460	0.460	0.460	0.000
Means	4	0.030	0.460	0.143	0.211

Table 9.4.1.4.2 Aluminum Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder (Figure 83)
 Property: Al
 Method: 1

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
14	2	0.0010	0.0012	0.0011	0.0001
Means	1	0.0011	0.0011	0.0011	0.0000

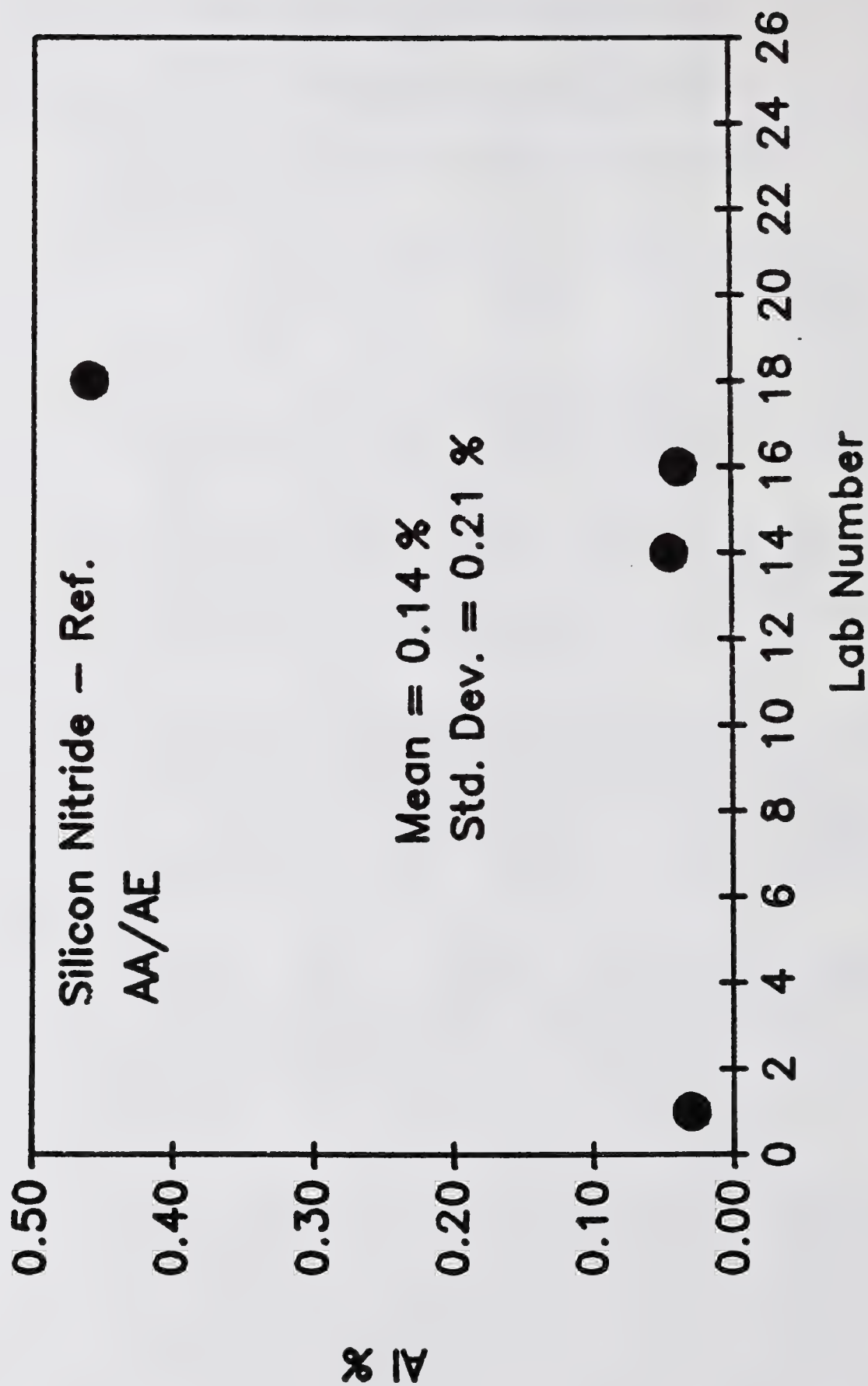


Figure 82. Aluminum concentration (wt. %) in SNR powder as reported by labs using AAS.

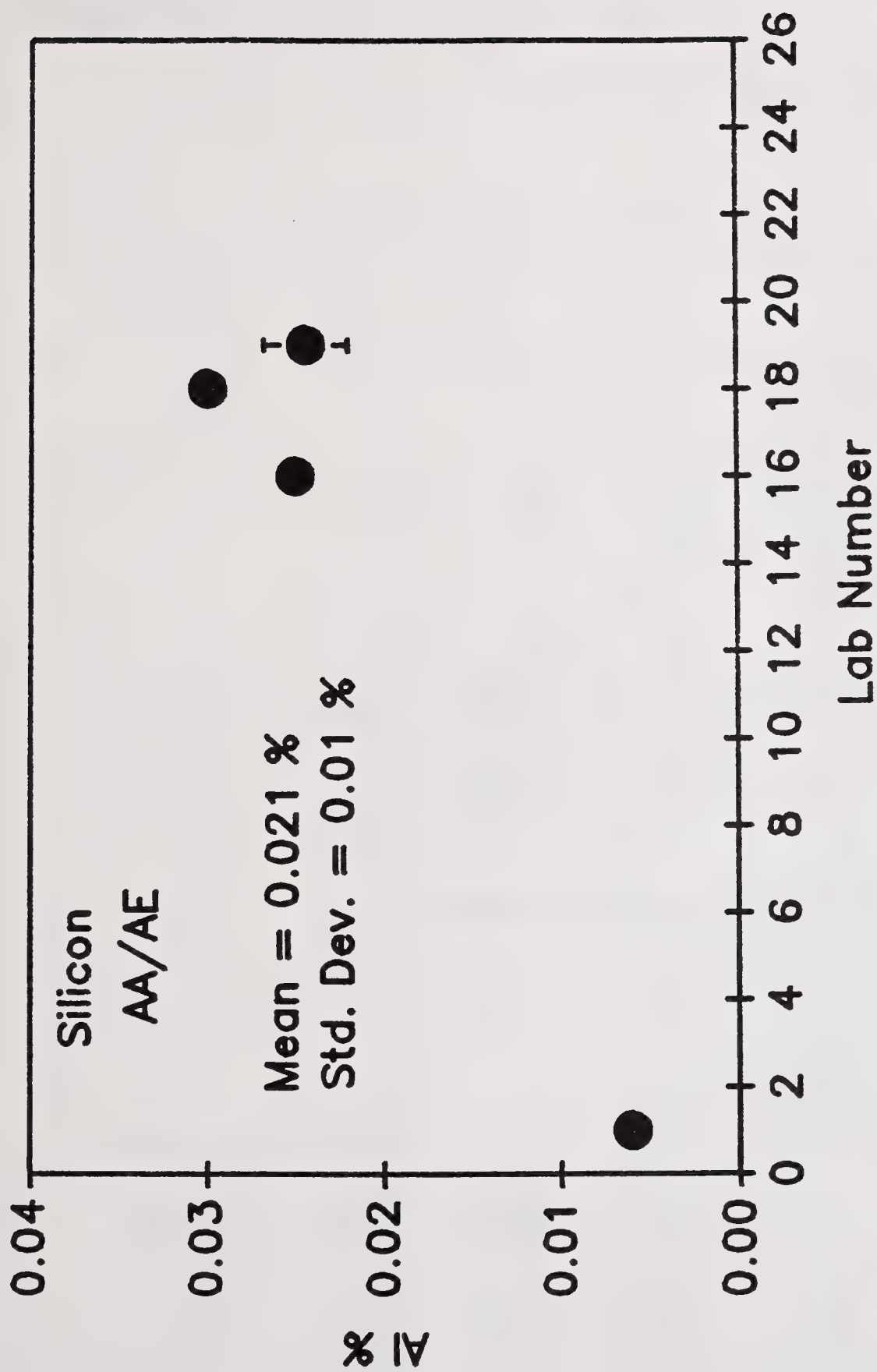


Figure 83. Aluminum concentration (wt. %) in Si powder as reported by labs using AAS.

Table 9.4.1.4.3 Aluminum Concentration (wt. %) in SiC Powder

Material: Silicon Carbide

Property: Al

Method: 1

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
16	2	0.025	0.026	0.025	0.0007
Means	1	0.025	0.025	0.025	0.0000

Table 9.4.1.4.4 Aluminum Concentration (wt. %) in Si Powder

Material: Silicon

Property: Al

Method: 1

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	1	0.006	0.006	0.006	0.000
16	2	0.025	0.025	0.025	0.000
18	1	0.030	0.030	0.030	0.000
19	9	0.021	0.028	0.024	0.002
Means	4	0.006	0.030	0.021	0.010

Table 9.4.1.4.5 Aluminum Concentration (wt. %) in YSZ Powder

Material: Zirconia

Property: Al

Method: 1

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
14	2	0.035	0.036	0.035	0.0007
19	8	0.070	0.080	0.071	0.0035
Means	2	0.035	0.071	0.053	0.0253

9.4.1. Aluminum in All Powders

9.4.1.5 Inductively Coupled Plasma - Emission Spectroscopy

Table 9.4.1.5.1 Aluminum Concentration (wt. %) in SNR Powder

Material: Silicon Nitride, Reference Powder (Figure 84)
 Property: Al
 Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	3	0.030	0.040	0.037	0.006
5	1	0.038	0.038	0.038	0.000
6	2	0.036	0.048	0.042	0.008
7	6	0.043	0.046	0.045	0.001
13	11	0.020	0.040	0.035	0.008
15	1	0.041	0.041	0.041	0.000
19	1	0.043	0.043	0.043	0.000
20	1	0.001	0.001	0.001	0.000
21	4	0.043	0.045	0.044	0.001
22	8	0.028	0.053	0.041	0.010
Means	10	0.0015	0.0450	0.0369	0.0128

Table 9.4.1.5.2 Aluminum Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder (Figure 85)
 Property: Al
 Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
5	4	0.000	0.000	0.000	0.000
17	1	0.504	0.504	0.504	0.000
18	1	0.200	0.200	0.200	0.000
19	7	0.000	0.000	0.000	0.000
20	1	0.000	0.000	0.000	0.000
22	8	0.016	0.022	0.020	0.002
Means	6	0.0001	0.504	0.120	0.203

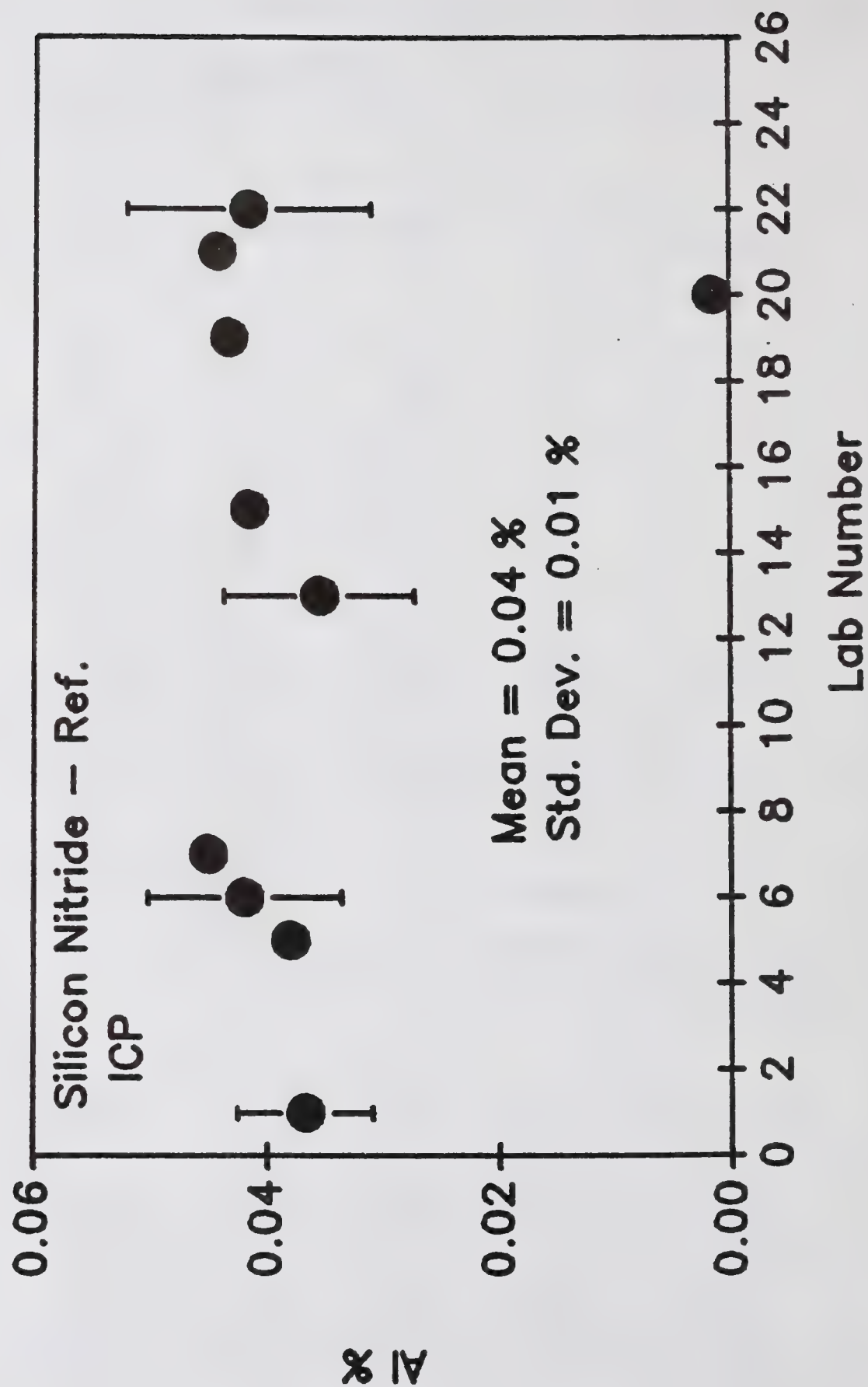


Figure 84. Aluminum concentration (wt. %) in SNR powder as reported by labs using ICP.

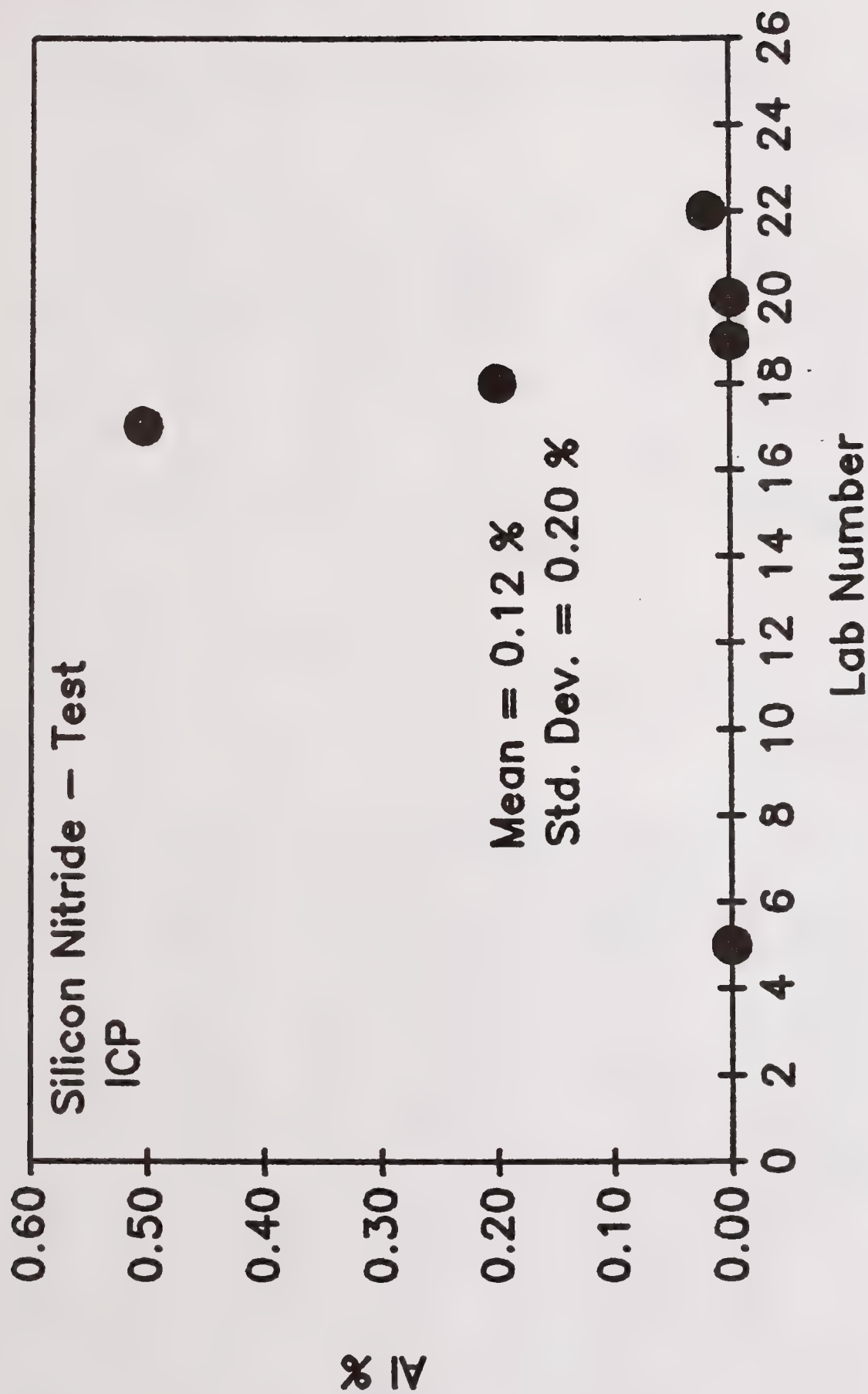


Figure 85. Aluminum concentration (wt. %) in SNT powder as reported by labs using ICP.

Table 9.4.1.5.3 Aluminum Concentration (wt. %) in SiC Powder

Material: Silicon Carbide (Figure 86)

Property: Al

Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
6	1	0.026	0.026	0.026	0.000
15	1	0.033	0.033	0.033	0.000
17	1	0.013	0.013	0.013	0.000
20	2	0.019	0.100	0.059	0.057
21	3	0.044	0.047	0.046	0.001
22	8	0.024	0.086	0.050	0.022
Means	6	0.013	0.059	0.038	0.017

Table 9.4.1.5.4 Aluminum Concentration (wt. %) in Si Powder

Material: Silicon (Figure 87)

Property: Al

Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
5	4	0.016	0.017	0.017	0.000
6	1	0.035	0.035	0.035	0.000
7	2	0.032	0.033	0.032	0.001
15	1	0.040	0.040	0.040	0.000
17	1	0.024	0.024	0.024	0.000
20	2	0.028	0.031	0.029	0.002
22	8	0.013	0.032	0.027	0.006
Means	7	0.017	0.040	0.029	0.007

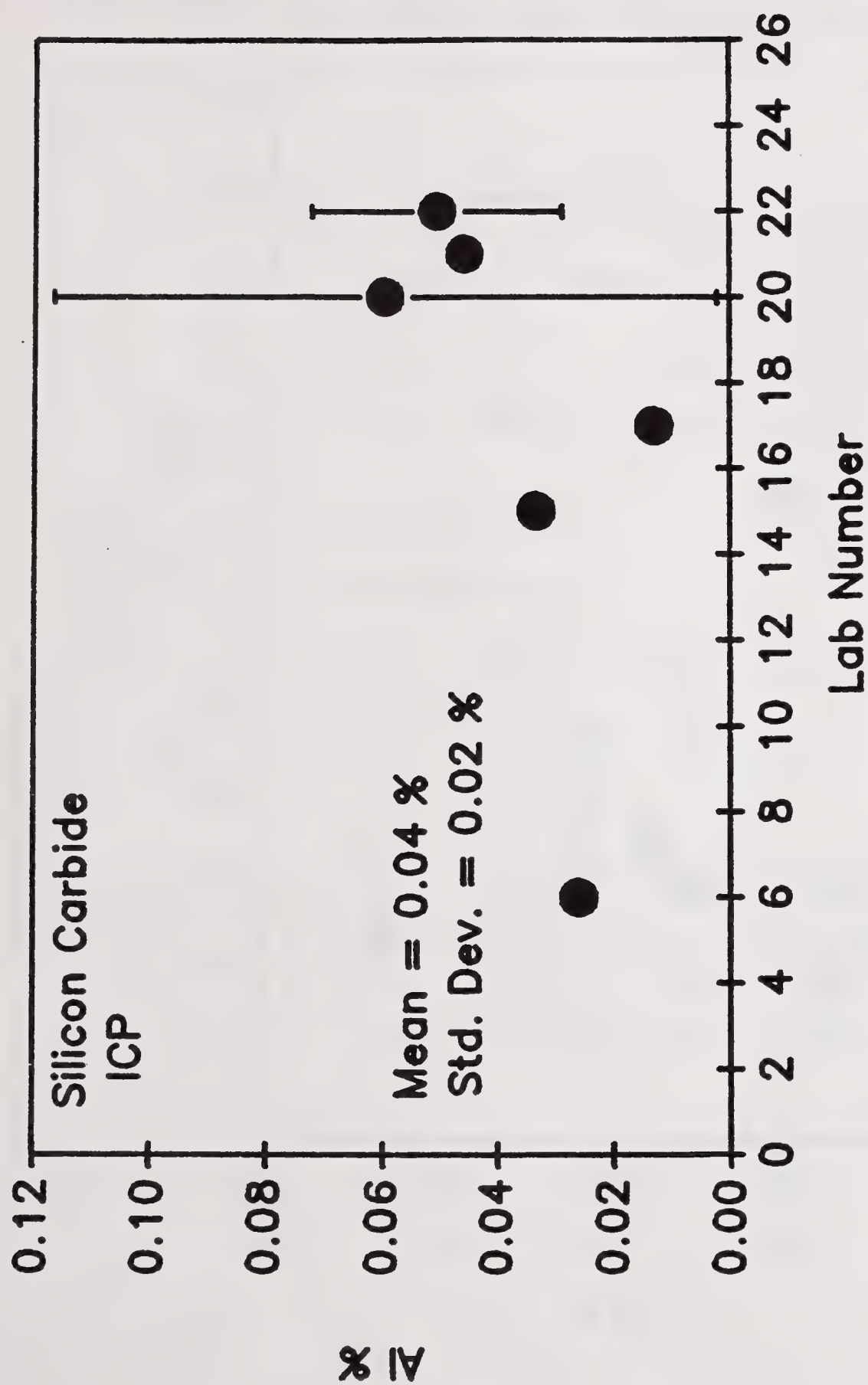


Figure 86. Aluminum concentration (wt. %) in SiC powder as reported by labs using ICP.

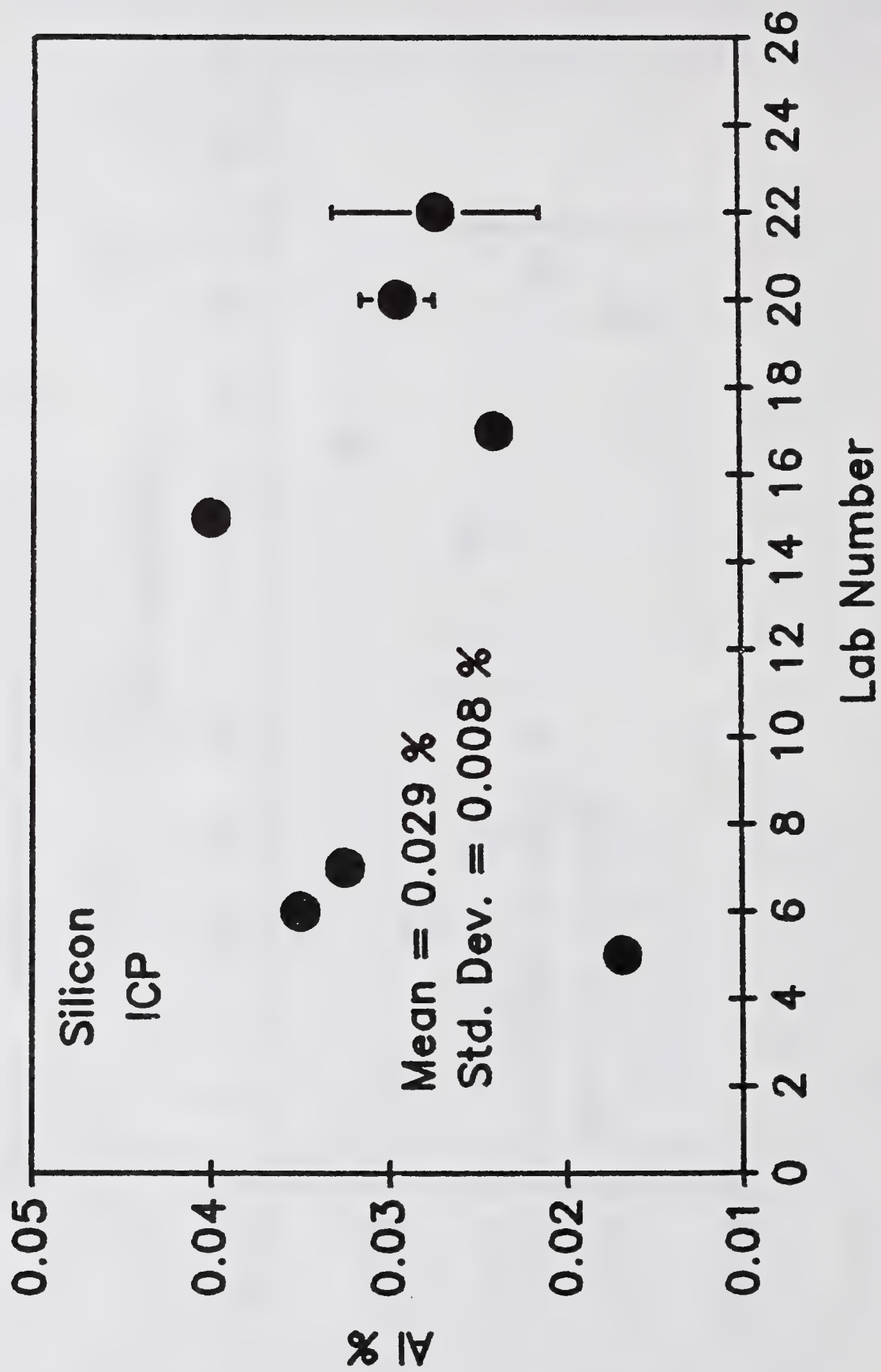


Figure 87. Aluminum concentration (wt. %) in Si powder as reported by labs using ICP.

Table 9.4.1.5.5 Aluminum Concentration (wt. %) in YSZ Powder

Material: Zirconia (Figure 88)

Property: Al

Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	2	0.040	0.050	0.045	0.007
6	2	0.066	0.314	0.190	0.175
15	1	0.052	0.052	0.052	0.000
17	1	0.260	0.260	0.260	0.000
Means	4	0.045	0.260	0.137	0.106

9.4.1. Aluminum in All Powder

9.4.1.6 Arc Emission Spectroscopy

Table 9.4.1.6.1 Aluminum Concentration (wt. %) in SNR Powder

Material: Silicon Nitride, Reference Powder

Property: Al

Method: 7

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
7	1	0.060	0.060	0.060	0.000
23	1	0.010	0.010	0.010	0.000
Means	2	0.010	0.060	0.035	0.035

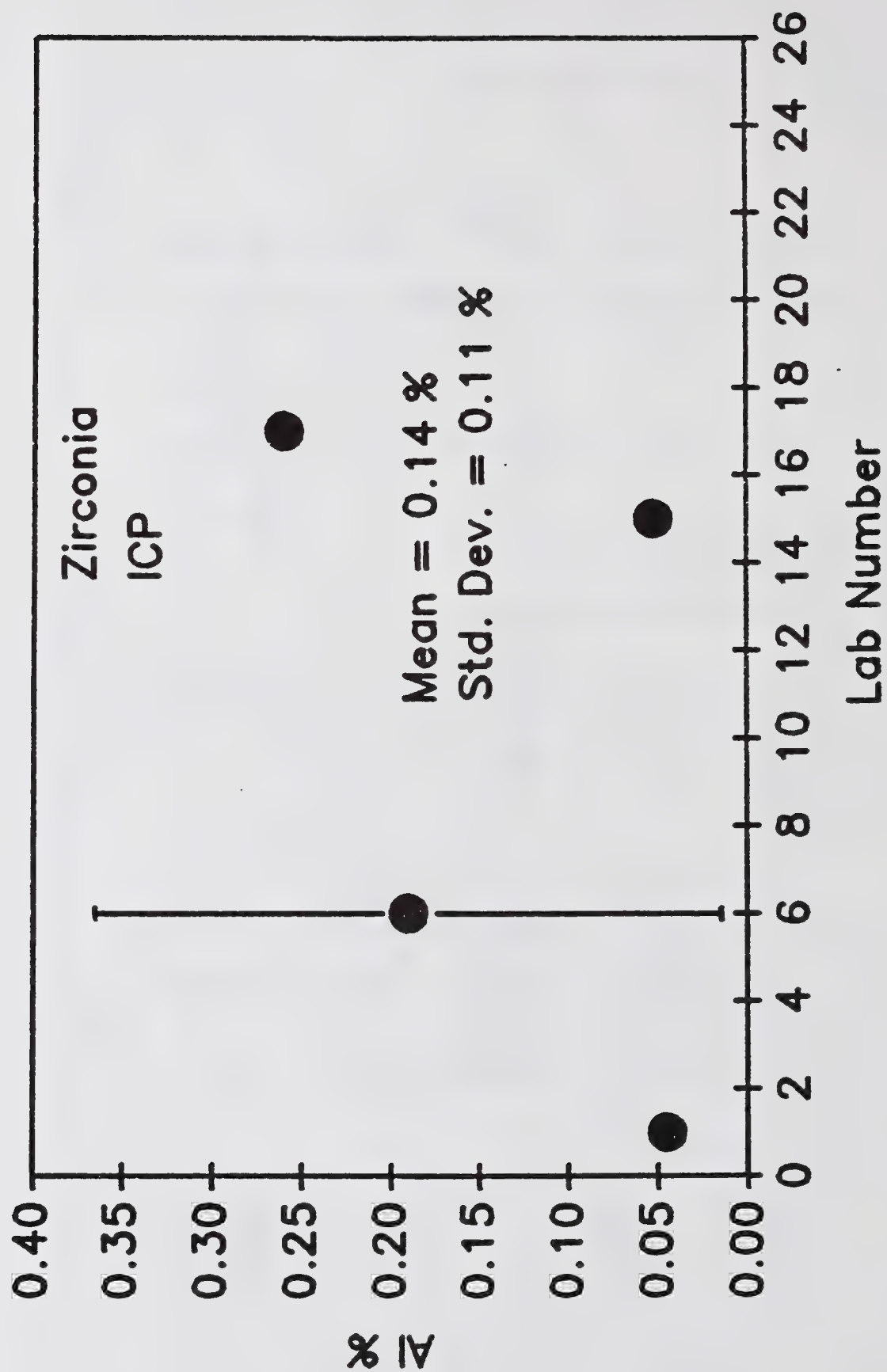


Figure 88. Aluminum concentration (wt. %) in YSZ powder as reported by labs using ICP.

Table 9.4.1.6.2 Aluminum Concentration (wt. %) in SiC Powder

Material: Silicon Carbide

Property: Al

Method: 7

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	1	0.010	0.010	0.010	0.000
Means	1	0.010	0.010	0.010	0.000

Table 9.4.1.6.3 Aluminum Concentration (wt. %) in Si Powder

Material: Silicon

Property: Al

Method: 7

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	1	0.020	0.020	0.020	0.000
Means	1	0.020	0.020	0.020	0.000

9.4.1. Aluminum in All Powder9.4.1.7 DC Plasma - Emission Spectroscopy

Table 9.4.1.7.1 Aluminum Concentration (wt. %) in SNR Powder

Material: Silicon Nitride, Reference Powder (Figure 89)

Property: Al

Method: 17

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
3	1	0.040	0.040	0.040	0.000
14	1	0.043	0.043	0.043	0.000
17	2	0.040	0.040	0.040	0.000
25	1	0.042	0.041	0.042	0.000
Means	4	0.040	0.043	0.041	0.001

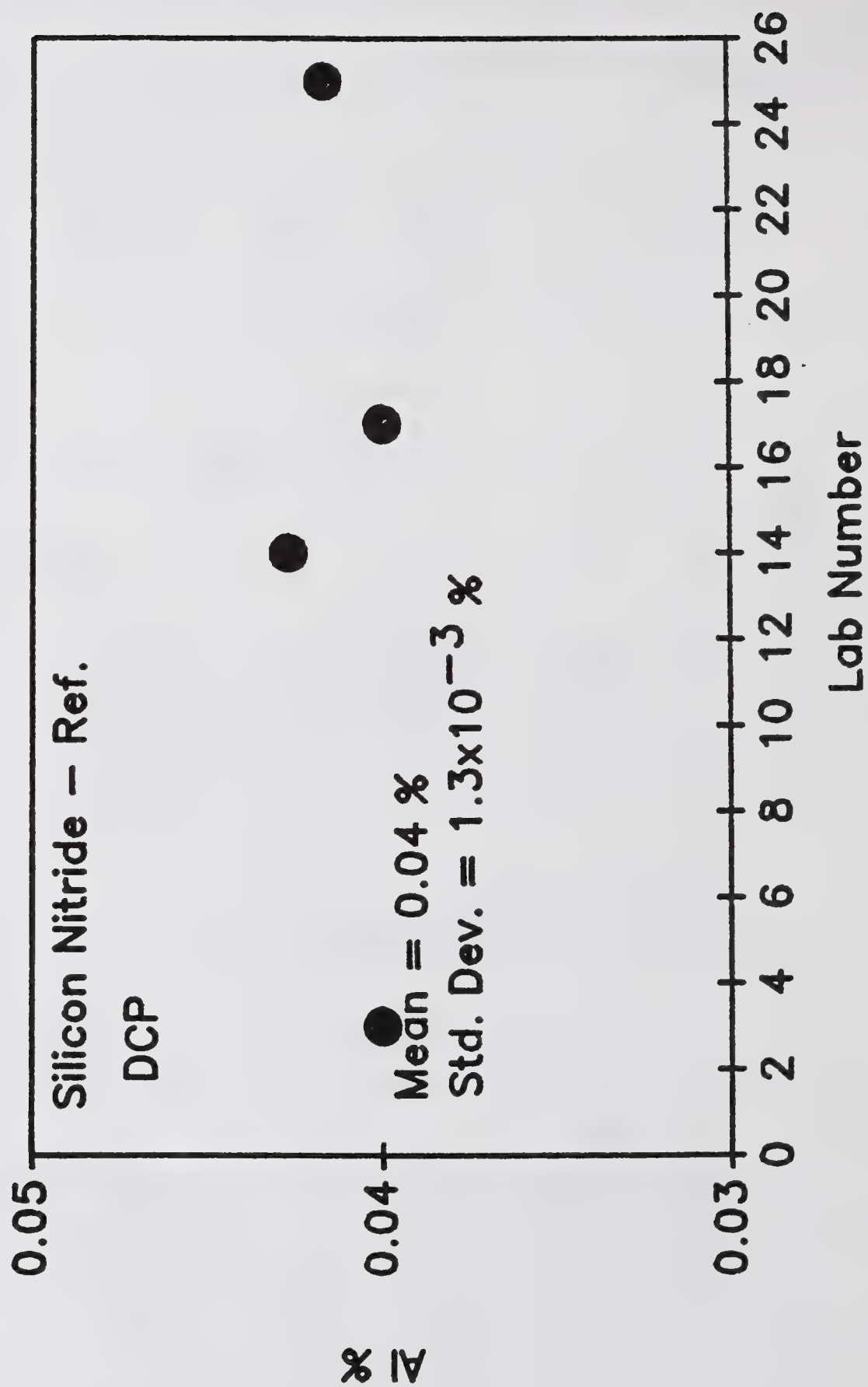


Figure 89. Aluminum concentration (wt. %) in SNR powder as reported by labs using DCP.

Table 9.4.1.7.2 Aluminum Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder

Property: Al

Method: 17

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
14	2	0.0015	0.0017	0.0016	0.0001
Means	1	0.0016	0.0016	0.0016	0.0000

Table 9.4.1.7.3 Aluminum Concentration (wt. %) in SiC Powder

Material: Silicon Carbide

Property: Al

Method: 17

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
25	6	0.0415	0.0464	0.0436	0.0019
Means	1	0.0436	0.0436	0.0436	0.0000

Table 9.4.1.7.4 Aluminum Concentration (wt. %) in Si Powder

Material: Silicon

Property: Al

Method: 17

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
3	4	0.0420	0.0440	0.0428	0.0010
Means	1	0.0428	0.0428	0.0428	0.0000

Table 9.4.1.7.5 Aluminum Concentration (wt. %) in YSZ Powder

Material: Zirconia

Property: Al

Method: 17

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
14	1	0.048	0.048	0.048	0.000
17	1	0.250	0.250	0.250	0.000
Means	2	0.048	0.250	0.149	0.143

9.4.1. Aluminum in All Powder

9.4.1.8 Method not specified

Table 9.4.1.8 Aluminum Concentration (wt. %) in SiC Powder

Material: Silicon Carbide

Property: Al

Method: 21

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
18	1	0.260	0.260	0.260	0.000
Means	1	0.260	0.260	0.260	0.000

9.4.2. Calcium in Silicon Nitride, Silicon Carbide and Yttria-Zirconia

9.4.2.1 Mass Spectrometry

Table 9.4.2.1.1 Calcium Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder
Property: Ca
Method: 5

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
8	3	0.0005	0.0016	0.0009	0.0006
Means	1	0.0009	0.0009	0.0009	0.0000

Table 9.4.2.1.2 Calcium Concentration (wt. %) in SiC Powder

Material: Silicon Carbide
Property: Ca
Method: 5

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
8	2	0.004	0.006	0.005	0.001
Means	1	0.005	0.005	0.005	0.000

Table 9.4.2.1.3 Calcium Concentration (wt. %) in YSZ Powder

Material: Zirconia
Property: Ca
Method: 5

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
8	3	0.002	0.007	0.0037	0.0029
Means	1	0.0037	0.0037	0.0037	0.000

9.4.2.2 Neutron Activation Analysis

Table 9.4.2.2 Calcium Concentration (wt. %) in YSZ Powder

Material: Zirconia
Property: Ca
Method: 6

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
2	1	0.027	0.027	0.027	0.000
Means	1	0.027	0.027	0.027	0.000

9.4.2.3 X-ray Fluorescence Spectrometry

Table 9.4.2.3.1 Calcium Concentration (wt. %) in SiC Powder

Material: Silicon Carbide
Property: Ca
Method: 12

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
10	1	0.002	0.002	0.002	0.000
Means	1	0.002	0.002	0.002	0.000

Table 9.4.2.3.2 Calcium Concentration (wt. %) in YSZ Powder

Material: Zirconia
Property: Ca
Method: 12

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
24	3	0.022	0.023	0.023	0.0006
Means	1	0.023	0.023	0.023	0.0000

9.4.2.4 Atomic Absorption Spectroscopy

Table 9.4.2.4.1 Calcium Concentration (wt. %) in SNR Powder

Material: Silicon Nitride, Reference Powder (Figure 90)
 Property: Ca
 Method: 1

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	1	0.020	0.020	0.020	0.000
14	2	0.020	0.020	0.020	0.000
16	6	0.006	0.011	0.008	0.002
18	1	0.020	0.020	0.020	0.000
24	3	0.050	0.098	0.072	0.024
Means	5	0.008	0.072	0.028	0.025

Table 9.4.2.4.2 Calcium Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder
 Property: Ca
 Method: 1

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
14	1	0.003	0.003	0.003	0.000
24	2	0.029	0.045	0.037	0.011
Means	2	0.003	0.037	0.020	0.023

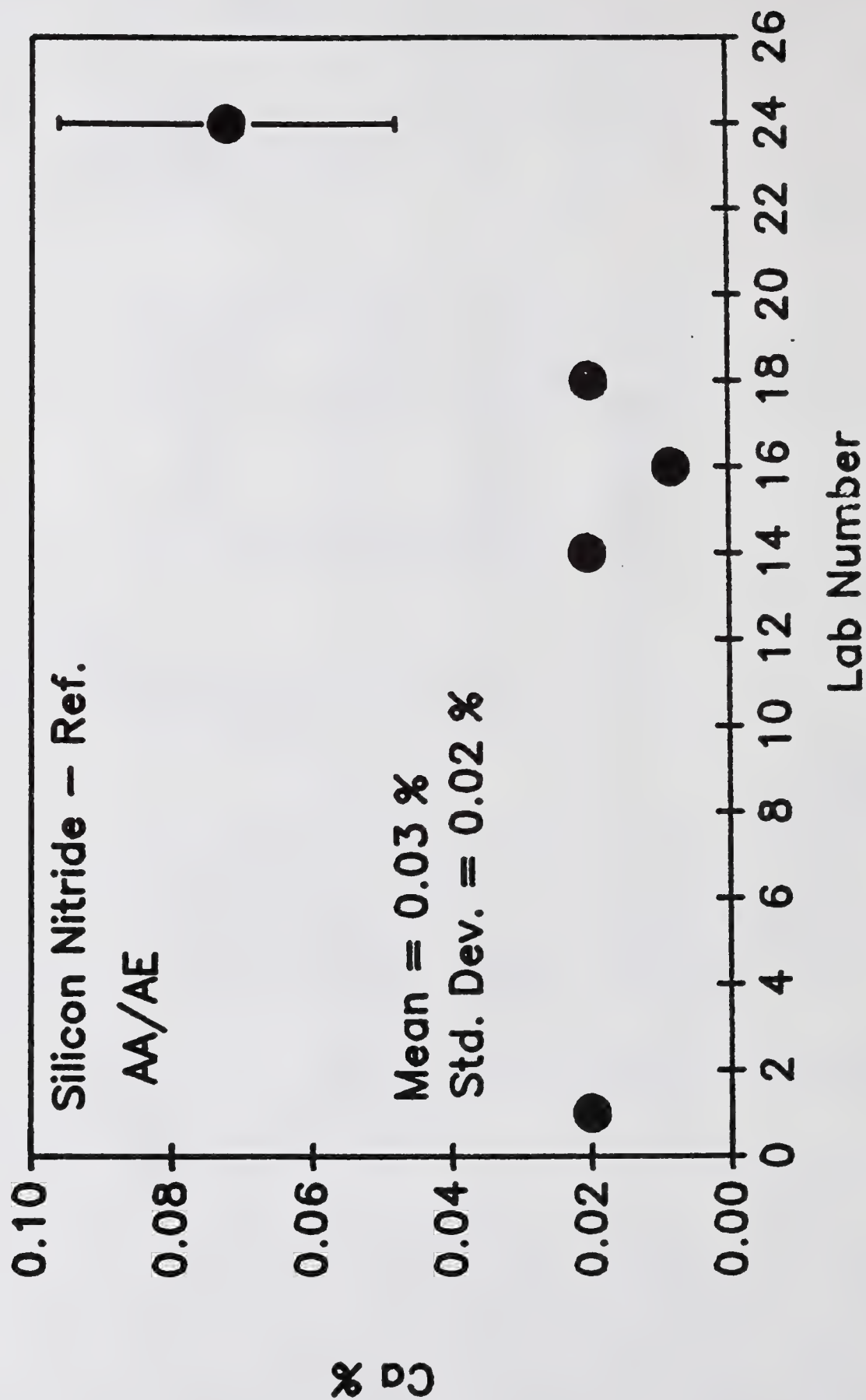


Figure 90. Calcium concentration (wt. %) in SNR powder as reported by labs using AAS.

Table 9.4.2.4.3 Calcium Concentration (wt. %) in SiC Powder

Material: Silicon Carbide
 Property: Ca
 Method: 1

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
10	1	0.014	0.014	0.014	0.000
16	2	0.006	0.006	0.006	0.000
Means	2	0.006	0.014	0.010	0.005

Table 9.4.2.4.4 Calcium Concentration (wt. %) in YSZ Powder

Material: Zirconia
 Property: Ca
 Method: 1

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
14	2	0.014	0.040	0.027	0.018
18	3	0.012	0.014	0.013	0.001
19	8	0.010	0.010	0.010	0.000
Means	3	0.010	0.027	0.017	0.009

9.4.2.5 Inductively Coupled Plasma - Emission Spectroscopy

Table 9.4.2.5.1 Calcium Concentration (wt. %) in SNR Powder

Material: Silicon Nitride, Reference Powder (Figure 91)
 Property: Ca
 Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	3	0.010	0.010	0.010	0.000
5	1	0.008	0.008	0.008	0.000
6	2	0.010	0.014	0.012	0.003
7	6	0.009	0.010	0.010	0.000
13	11	0.010	0.040	0.015	0.009
15	1	0.009	0.009	0.009	0.000
19	1	0.009	0.009	0.009	0.000
20	1	0.001	0.001	0.001	0.000
21	4	0.011	0.012	0.011	0.000
22	11	0.001	0.012	0.005	0.004
Means	10	0.0015	0.015	0.009	0.004

Table 9.4.2.5.2 Calcium Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder (Figure 92)
 Property: Ca
 Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
5	4	0.000	0.000	0.000	0.000
6	1	0.007	0.007	0.007	0.000
15	1	0.000	0.000	0.000	0.000
17	1	0.012	0.012	0.012	0.000
18	1	0.010	0.010	0.010	0.000
19	7	0.000	0.000	0.000	0.000
20	1	0.000	0.000	0.000	0.000
21	4	0.001	0.001	0.001	0.000
22	2	0.001	0.002	0.001	0.001
Means	9	0.000	0.012	0.004	0.004

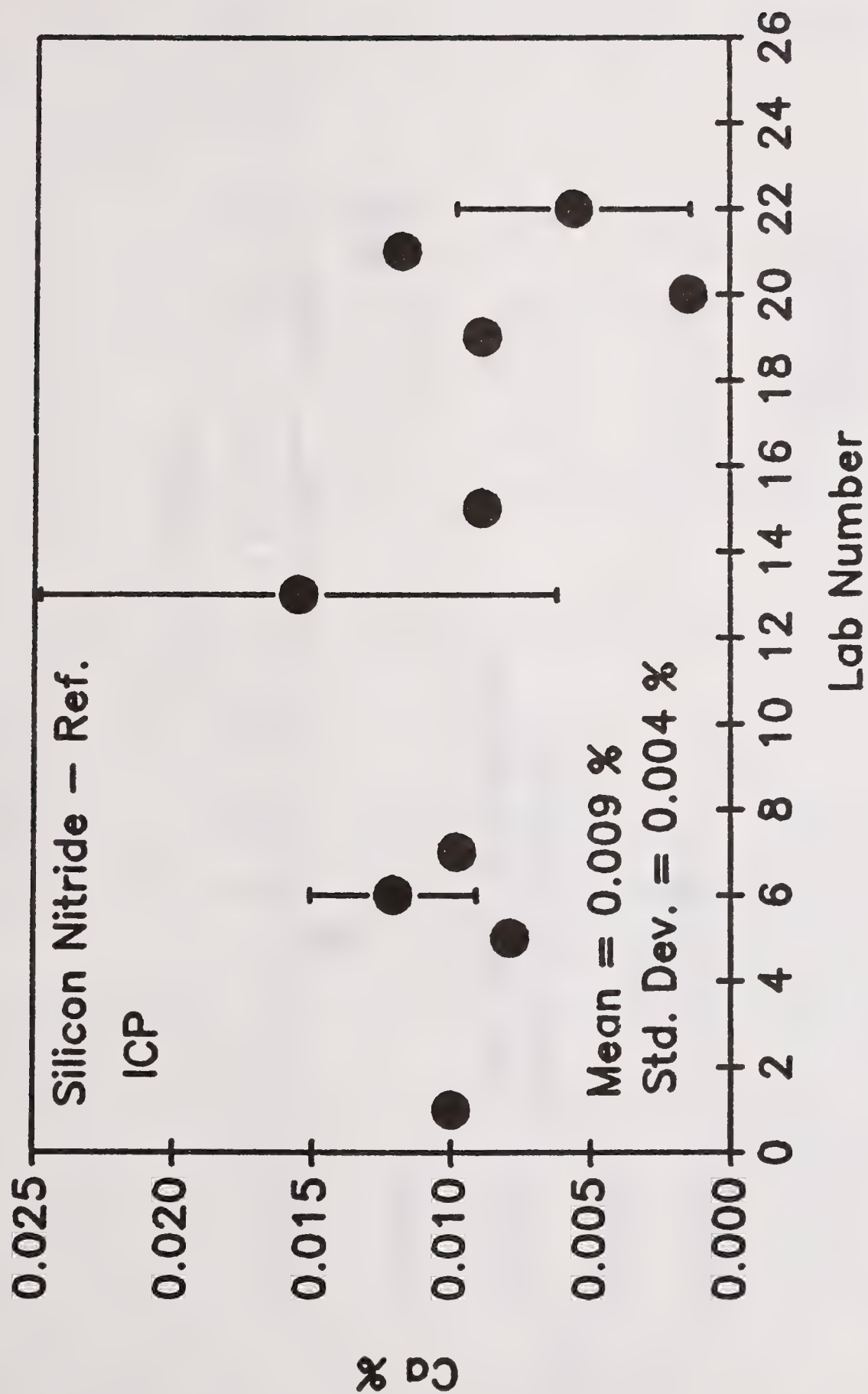


Figure 91. Calcium concentration (wt. %) in SNR powder as reported by labs using ICP.

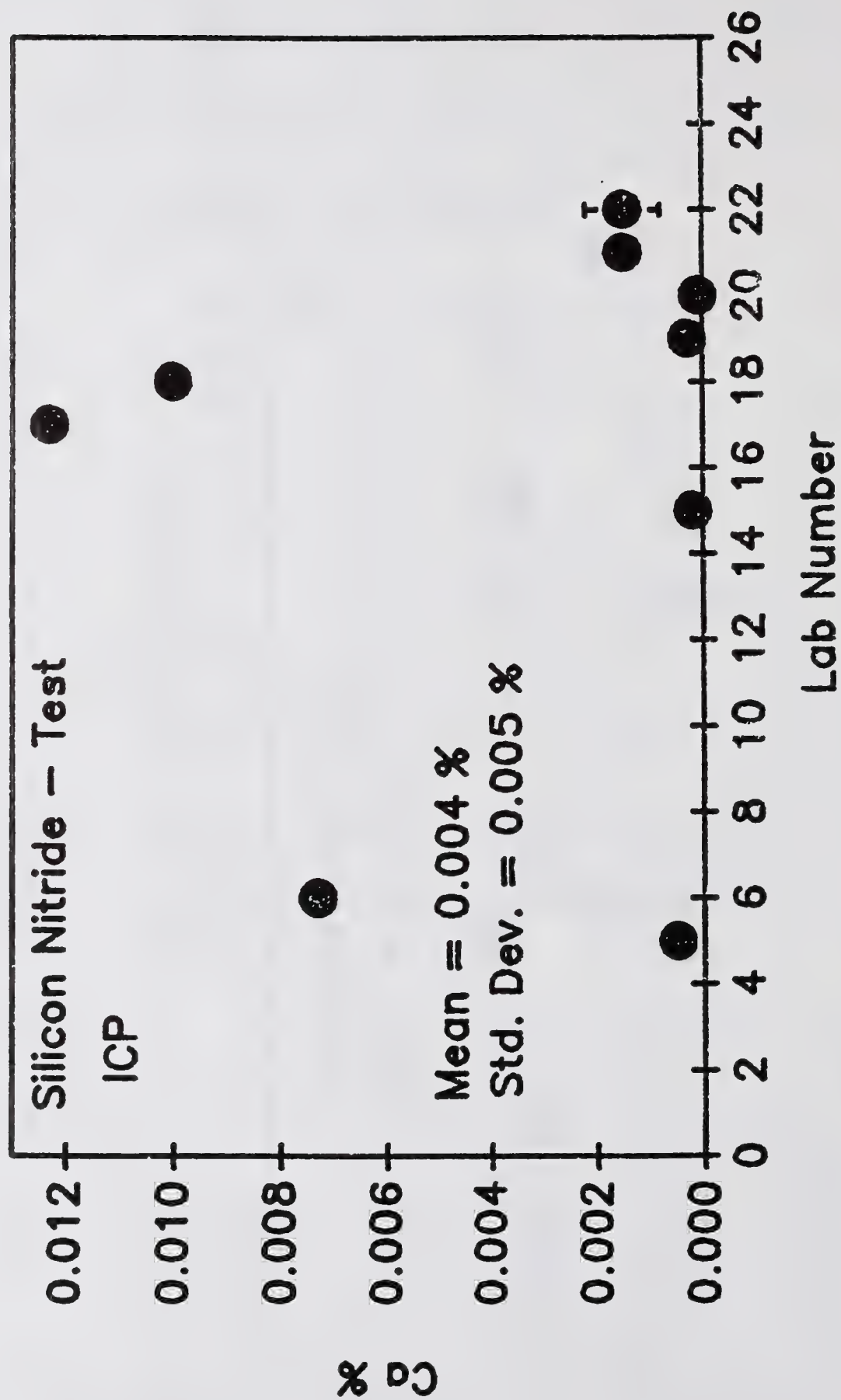


Figure 92. Calcium concentration (wt. %) in SNT powder as reported by labs using ICP.

Table 9.4.2.5.3 Calcium Concentration (wt. %) in SiC Powder

Material: Silicon Carbide (Figure 93)

Property: Ca

Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
6	1	0.010	0.010	0.010	0.000
15	1	0.008	0.008	0.008	0.000
17	1	0.007	0.007	0.007	0.000
20	2	0.120	0.150	0.135	0.021
21	3	0.006	0.006	0.006	0.000
22	8	0.001	0.027	0.013	0.010
Means	6	0.006	0.135	0.030	0.051

Table 9.4.2.5.4 Calcium Concentration (wt. %) in YSZ Powder

Material: Zirconia

Property: Ca

Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
6	1	0.034	0.034	0.034	0.000
15	1	0.011	0.011	0.011	0.000
17	1	0.020	0.020	0.020	0.000
Means	3	0.011	0.034	0.021	0.011

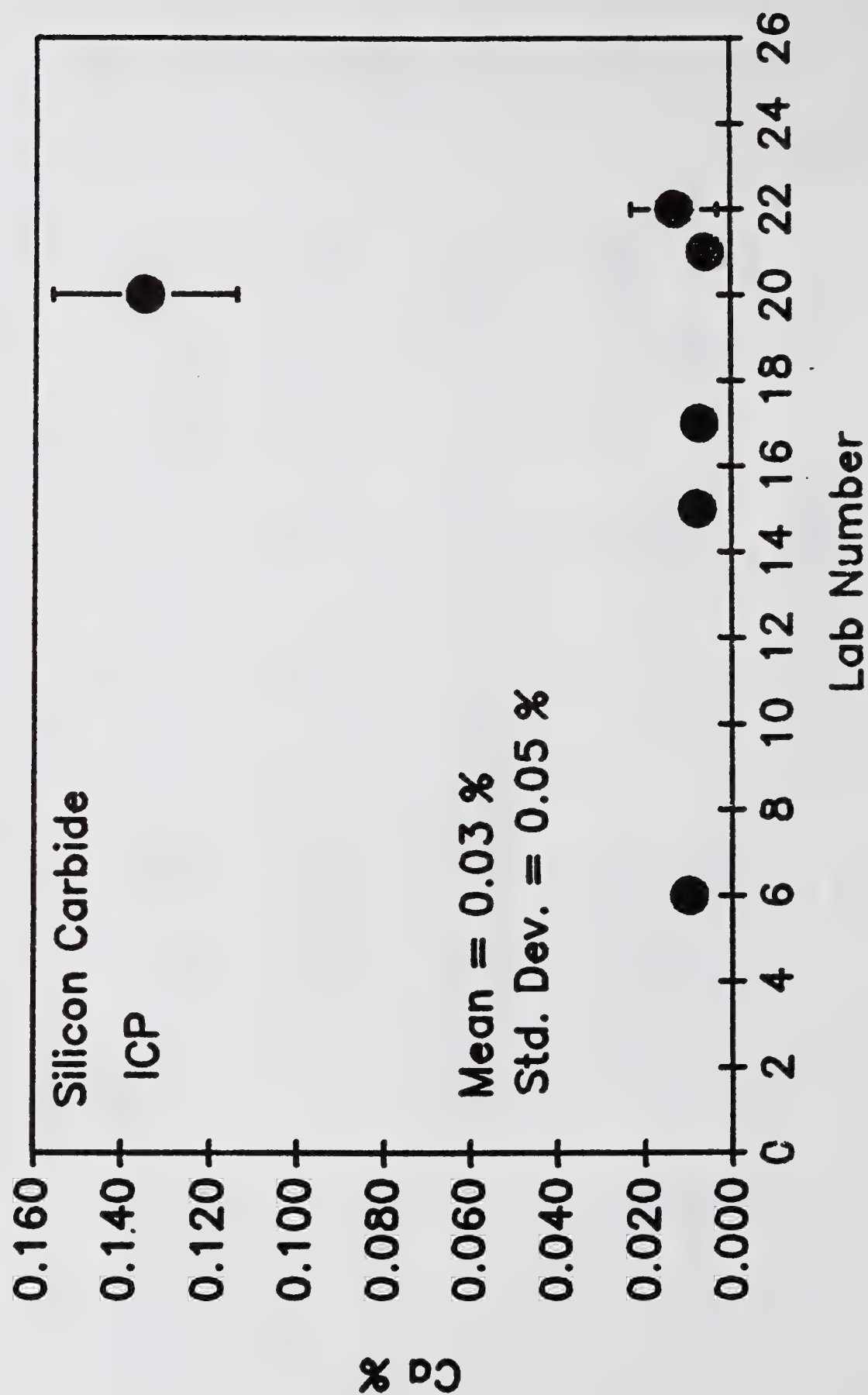


Figure 93. Calcium concentration (wt. %) in SiC powder as reported by labs using ICP.

9.4.2.6 Arc Emission Spectroscopy

Table 9.4.2.6 Calcium Concentration (wt. %) in SiC Powder

Material: Silicon Carbide
Property: Ca
Method: 7

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	1	0.010	0.010	0.010	0.000
Means	1	0.010	0.010	0.010	0.000

9.4.2.7 DC Plasma - Emission Spectroscopy

Table 9.4.2.7.1 Calcium Concentration (wt. %) in SNR Powder

Material: Silicon Nitride, Reference Powder (Figure 94)
Property: Ca
Method: 17

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
3	1	0.030	0.030	0.030	0.000
14	1	0.008	0.008	0.008	0.000
17	2	0.007	0.010	0.008	0.002
25	1	0.009	0.009	0.009	0.000
Means	4	0.008	0.030	0.014	0.010

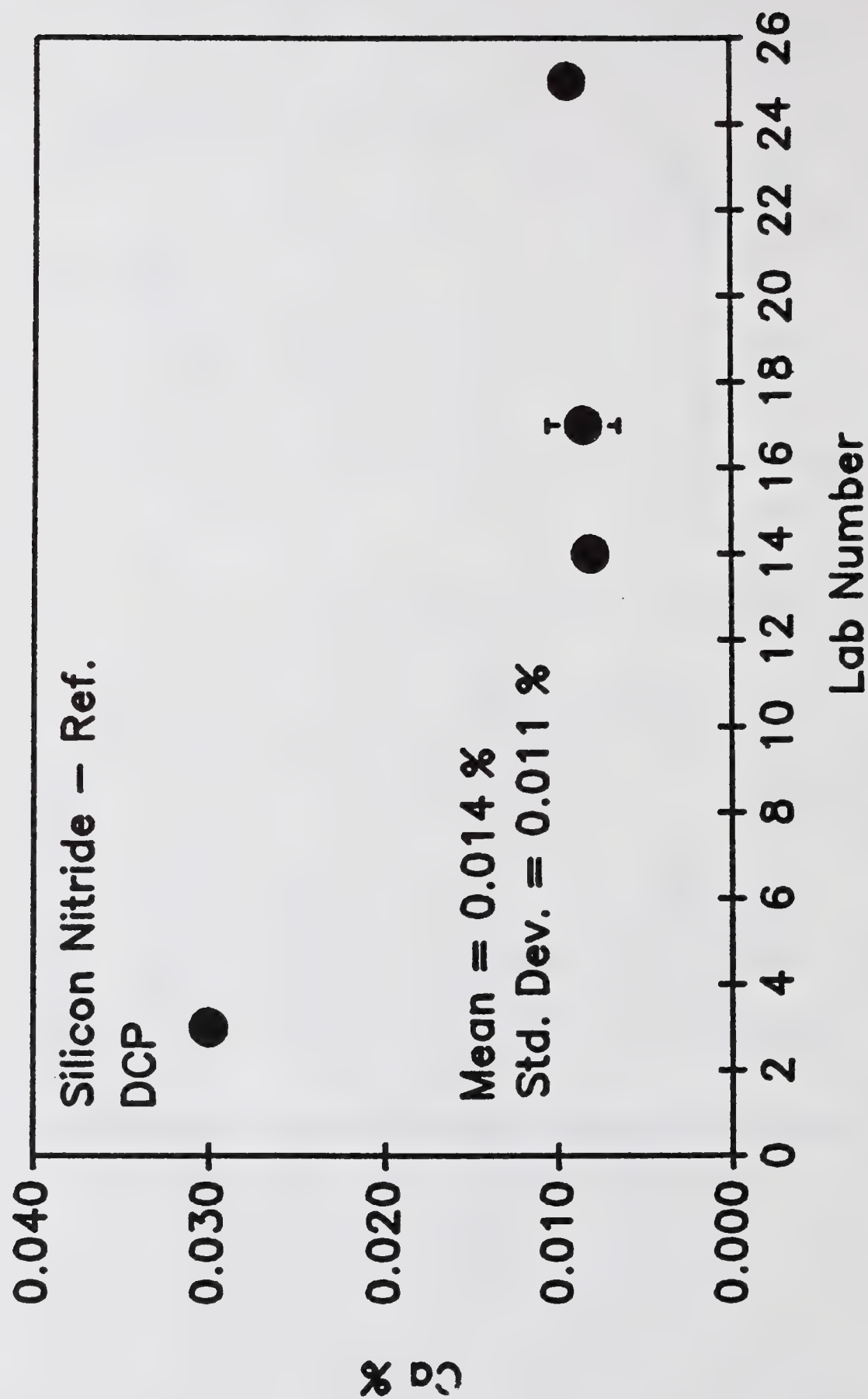


Figure 94. Calcium concentration (wt. %) in SNR powder as reported by labs using DCP.

Table 9.4.2.7.2 Calcium Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder

Property: Ca

Method: 17

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
14	2	0.003	0.003	0.003	0.000
Means	1	0.003	0.003	0.003	0.000

Table 9.4.2.7.3 Calcium Concentration (wt. %) in SiC Powder

Material: Silicon Carbide

Property: Ca

Method: 17

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
25	6	0.007	0.009	0.008	0.001
Means	1	0.008	0.008	0.008	0.000

Table 9.4.2.7.4 Calcium Concentration (wt. %) in YSZ Powder

Material: Zirconia

Property: Ca

Method: 17

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
17	1	0.020	0.020	0.020	0.000
Means	1	0.020	0.020	0.020	0.000

9.4.3 Iron in All Powders

9.4.3.1 Mass Spectrometry

Table 9.4.3.1.1 Iron Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder

Property: Fe

Method: 5

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
8	3	0.002	0.008	0.006	0.003
Means	1	0.006	0.006	0.006	0.000

Table 9.4.3.1.2 Iron Concentration (wt. %) in YSZ Powder

Material: Zirconia

Property: Fe

Method: 5

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
8	3	0.060	0.100	0.087	0.023
Means	1	0.087	0.087	0.087	0.000

9.4.3.2 Neutron Activation Analysis

Table 9.4.3.2 Iron Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder
 Property: Fe
 Method: 6

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
2	1	0.0037	0.0037	0.0037	0.000
Means	1	0.0037	0.0037	0.0037	0.000

9.4.3.3 X-ray Fluorescence Spectrometry

Table 9.4.3.3.1 Iron Concentration (wt. %) in SiC Powder

Material: Silicon Carbide
 Property: Fe
 Method: 12

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
10	1	0.014	0.014	0.014	0.000
Means	1	0.014	0.014	0.014	0.000

Table 9.4.3.3.2 Iron Concentration (wt. %) in YSZ Powder

Material: Zirconia

Property: Fe

Method: 12

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
24	3	0.014	0.015	0.014	0.000
Means	1	0.014	0.014	0.014	0.000

9.4.3.4 Atomic Absorption Spectroscopy

Table 9.4.3.4.1 Iron Concentration (wt. %) in SNR Powder

Material: Silicon Nitride, Reference Powder (Figure 95)

Property: Fe

Method: 1

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	1	0.010	0.010	0.010	0.000
14	2	0.015	0.015	0.015	0.000
16	6	0.019	0.026	0.021	0.002
18	1	0.270	0.270	0.270	0.000
24	3	0.030	0.035	0.031	0.003
Means	5	0.010	0.270	0.069	0.112

Table 9.4.3.4.2 Iron Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder

Property: Fe

Method: 1

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
14	2	0.005	0.005	0.005	0.000
16	1	0.004	0.004	0.004	0.000
24	2	0.030	0.035	0.032	0.003
Means	3	0.004	0.033	0.014	0.016

Table 9.4.3.4.3 Iron Concentration (wt. %) in SiC Powder

Material: Silicon Carbide

Property: Fe

Method: 1

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
10	1	0.001	0.001	0.001	0.000
16	2	0.001	0.002	0.002	0.000
Means	2	0.001	0.001	0.001	0.000

Table 9.4.3.4.4 Iron Concentration (wt. %) in Si Powder

Material: Silicon (Figure 96)

Property: Fe

Method: 1

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	1	0.045	0.045	0.045	0.000
16	2	0.038	0.038	0.038	0.000
18	1	0.050	0.050	0.050	0.000
19	9	0.042	0.047	0.043	0.001
Means	4	0.038	0.050	0.044	0.005

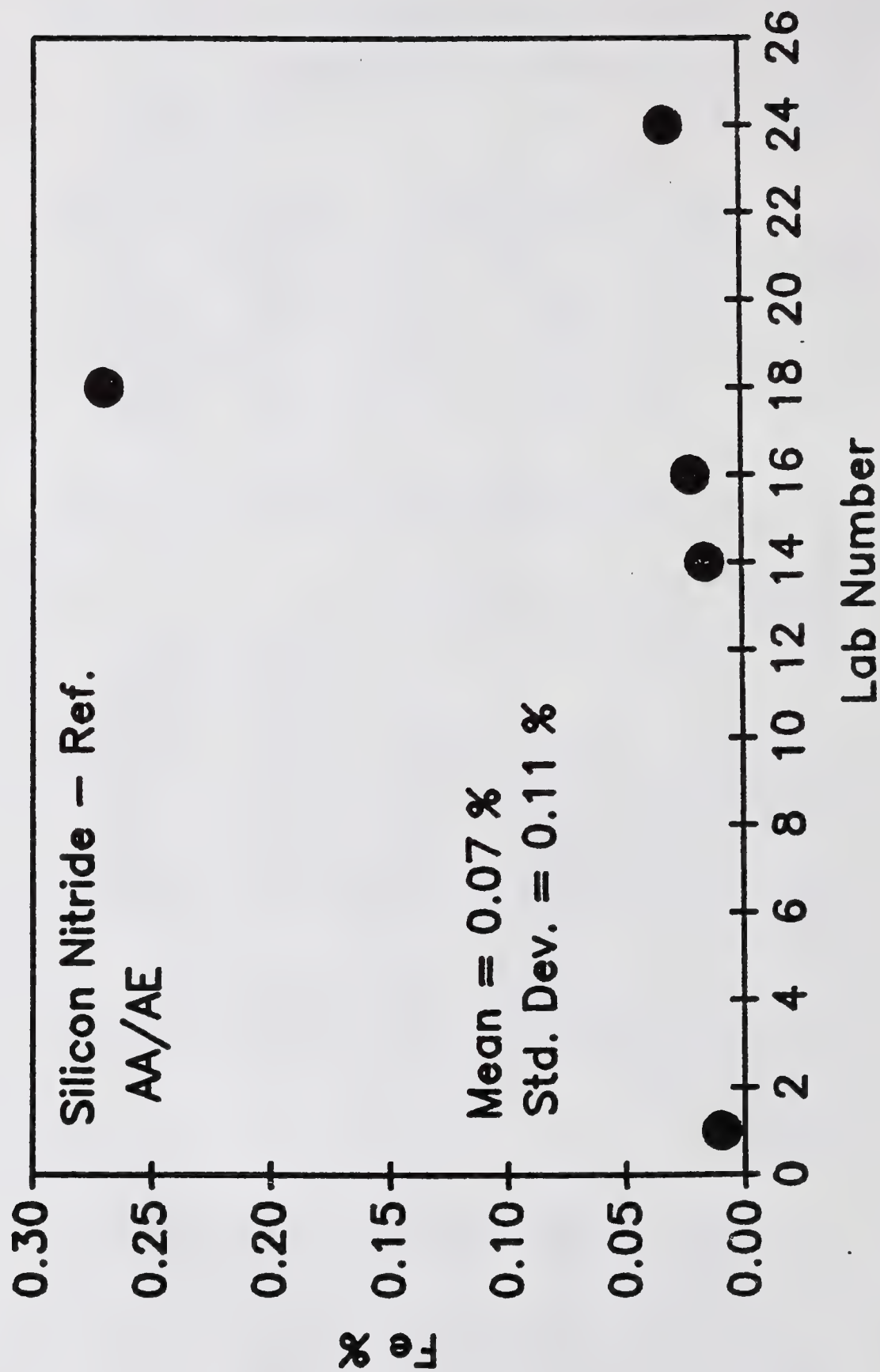


Figure 95. Iron concentration (wt. %) in SNR powder as reported by labs using AAS.

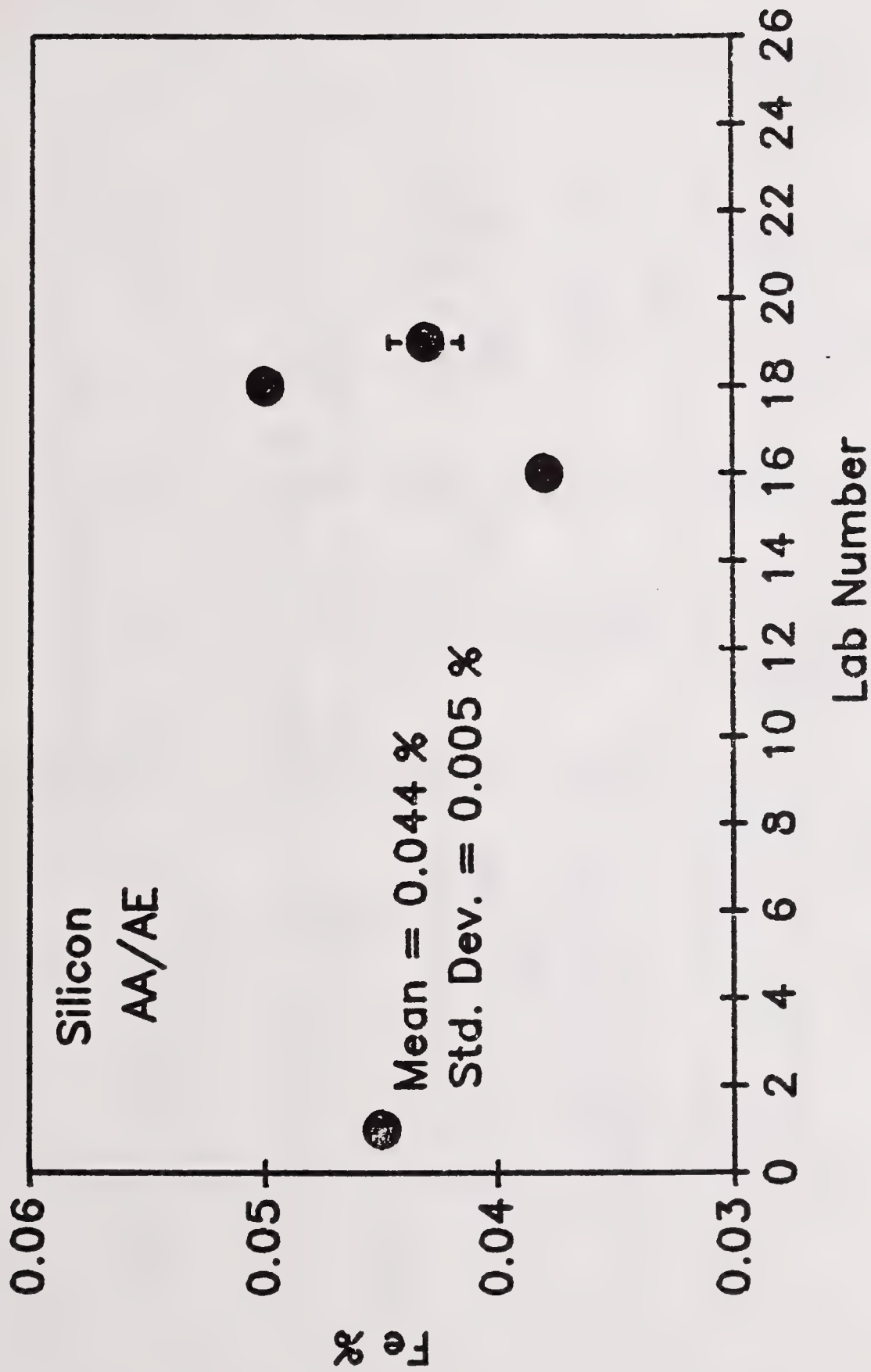


Figure 96. Iron concentration (wt. %) in Si powder as reported by labs using AAS.

Table 9.4.3.4.5 Iron Concentration (wt. %) in YSZ Powder

Material: Zirconia
 Property: Fe
 Method: 1

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
18	3	0.120	0.150	0.133	0.015
19	8	0.010	0.010	0.010	0.000
Means	2	0.010	0.133	0.071	0.087

9.4.3.5 Inductively Coupled Plasma - Emission Spectroscopy

Table 9.4.3.5.1 Iron Concentration (wt. %) in SNR Powder

Material: Silicon Nitride, Reference Powder (Figure 97)
 Property: Fe
 Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	1	0.010	0.010	0.010	0.000
5	1	0.018	0.018	0.018	0.000
6	2	0.019	0.049	0.034	0.021
7	6	0.021	0.023	0.022	0.001
13	7	0.010	0.010	0.010	0.000
15	1	0.021	0.021	0.021	0.000
19	1	0.020	0.020	0.020	0.000
20	1	0.006	0.006	0.006	0.000
21	4	0.023	0.024	0.024	0.000
22	11	0.011	0.029	0.021	0.008
Means	10	0.006	0.034	0.019	0.008

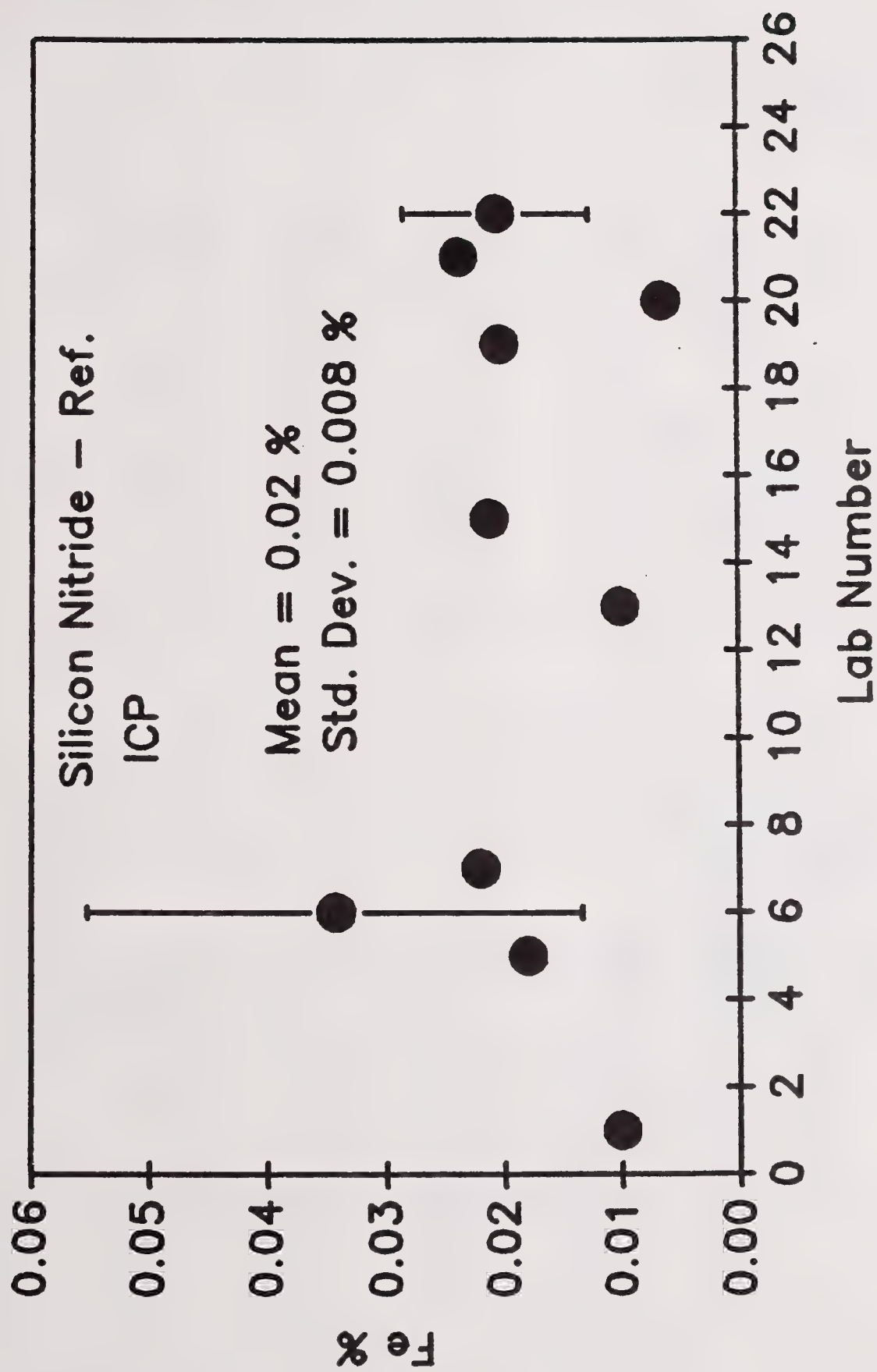


Figure 97. Iron concentration (wt. %) in SNR powder as reported by labs using ICP.

Table 9.4.3.5.2 Iron Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder (Figure 98)

Property: Fe

Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
5	4	0.003	0.003	0.003	0.000
6	1	0.005	0.005	0.005	0.000
7	3	0.002	0.003	0.003	0.000
15	1	0.004	0.004	0.004	0.000
17	1	0.019	0.019	0.019	0.000
18	1	0.200	0.200	0.200	0.000
19	7	0.004	0.004	0.004	0.000
20	1	0.000	0.001	0.001	0.000
21	4	0.004	0.005	0.005	0.000
22	8	0.010	0.011	0.010	0.000
Means	10	0.001	0.200	0.025	0.061

Table 9.4.3.5.3 Iron Concentration (wt. %) in SiC Powder

Material: Silicon Carbide (Figure 99)

Property: Fe

Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
6	1	0.004	0.004	0.004	0.000
15	1	0.005	0.005	0.005	0.000
20	2	0.060	0.190	0.125	0.092
21	3	0.001	0.001	0.001	0.000
22	8	0.004	0.013	0.009	0.004
Means	5	0.001	0.125	0.029	0.054

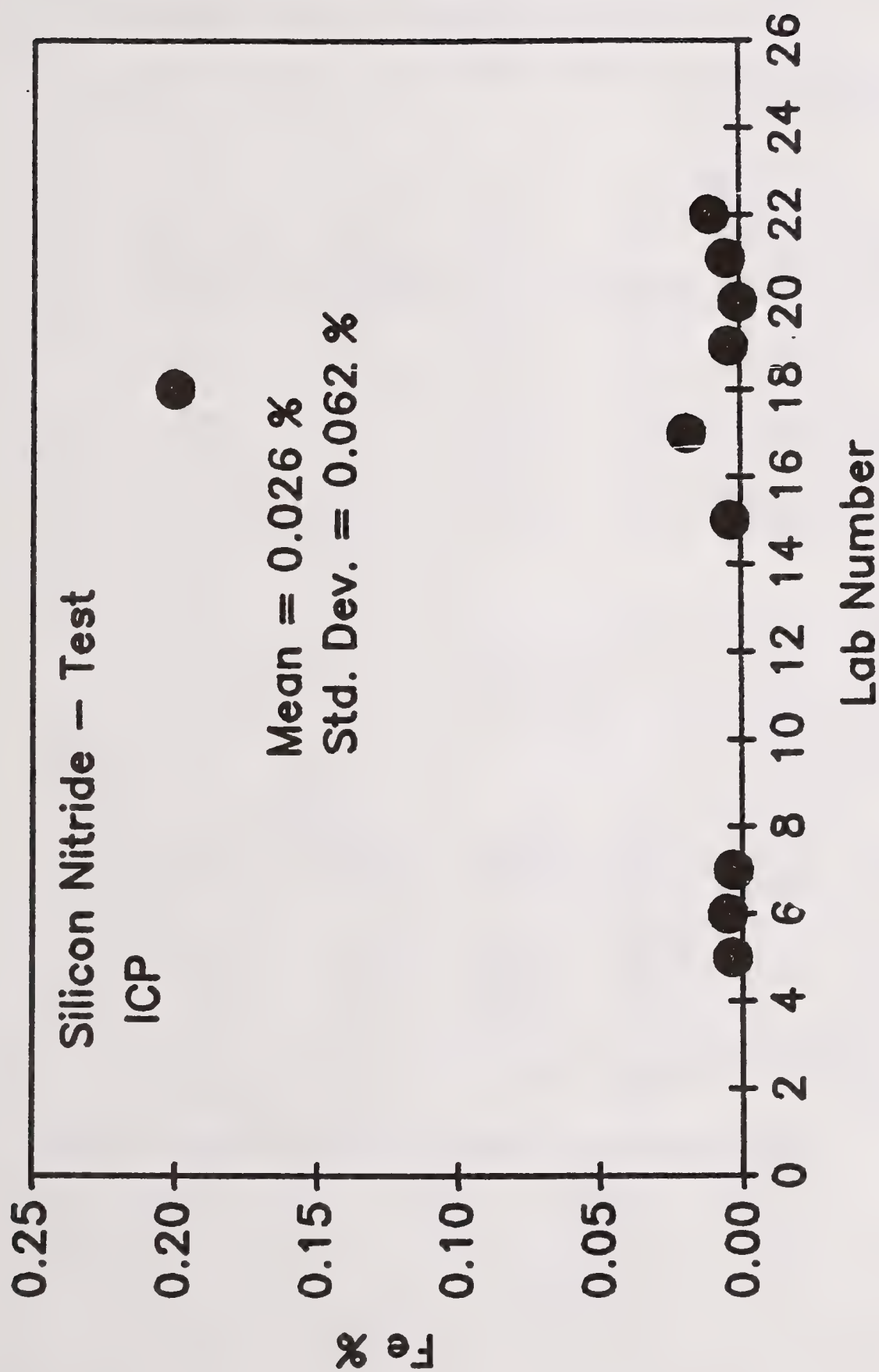


Figure 98. Iron concentration (wt. %) in SNT powder as reported by labs using ICP.

Table 9.4.3.5.4 Iron Concentration (wt. %) in Si Powder

Material: Silicon (Figure 100)

Property: Fe

Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
5	4	0.034	0.036	0.035	0.001
6	1	0.046	0.046	0.046	0.000
7	2	0.047	0.049	0.048	0.001
15	1	0.040	0.040	0.040	0.000
17	1	0.040	0.040	0.040	0.000
20	2	0.040	0.042	0.041	0.001
22	8	0.050	0.054	0.052	0.002
Means	7	0.035	0.052	0.043	0.006

Table 9.4.3.5.5 Iron Concentration (wt. %) in YSZ Powder

Material: Zirconia (Figure 101)

Property: Fe

Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	2	0.003	0.004	0.003	0.001
6	1	0.002	0.002	0.002	0.000
15	1	0.005	0.005	0.005	0.000
17	1	0.020	0.020	0.020	0.000
Means	4	0.002	0.020	0.008	0.008

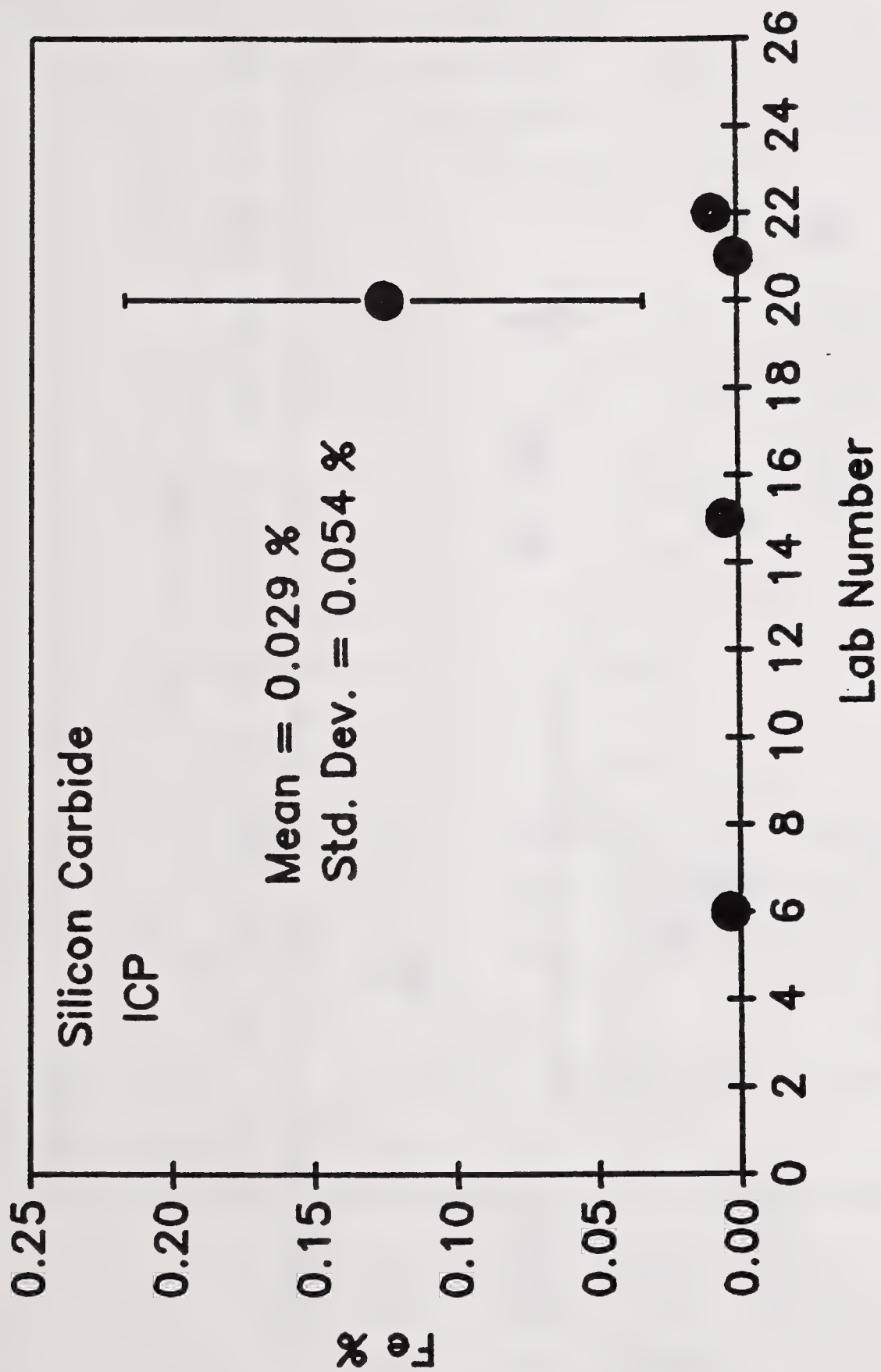


Figure 99. Iron concentration (wt. %) in SiC powder as reported by labs using ICP.

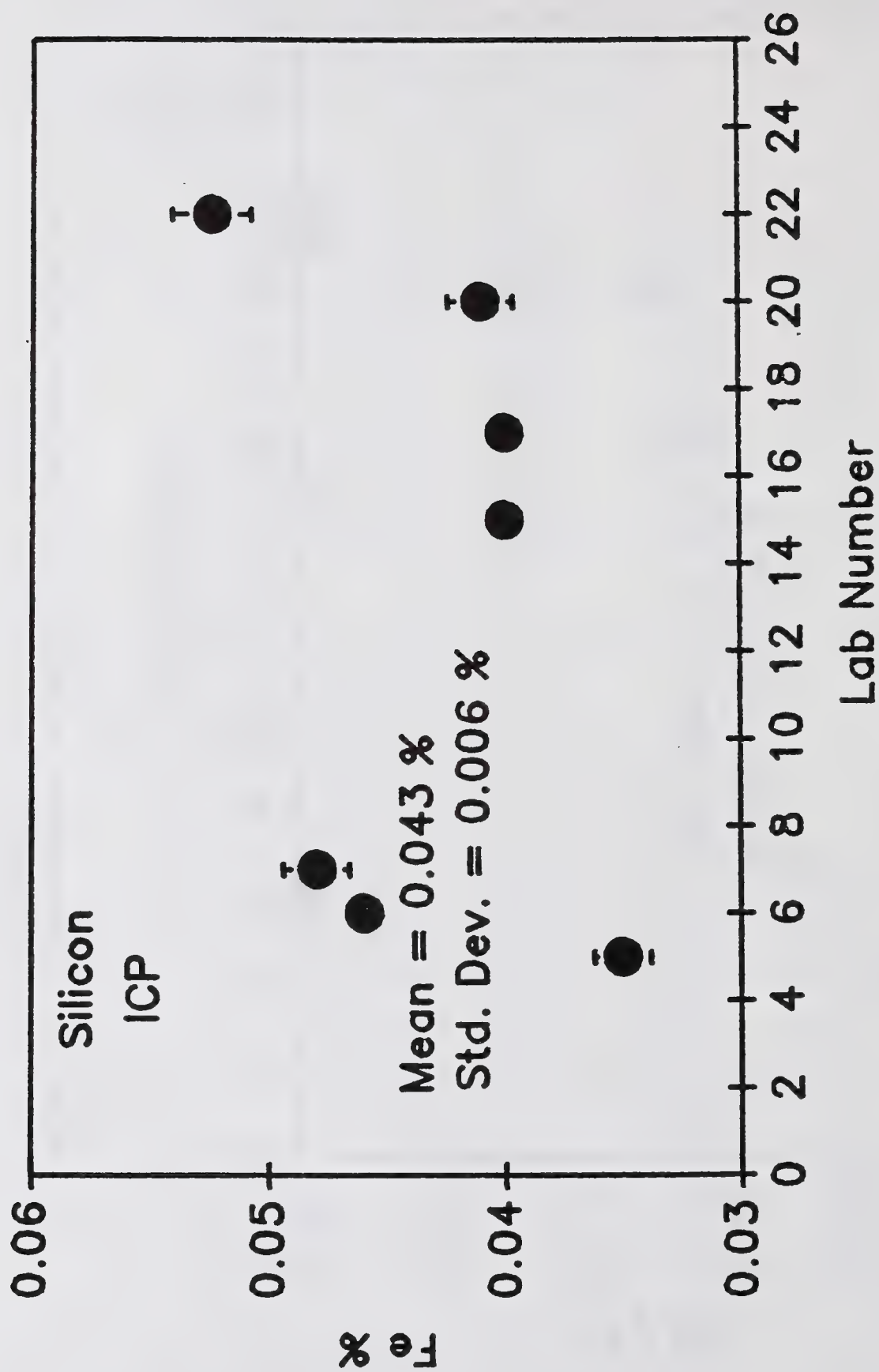


Figure 100. Iron concentration (wt. %) in Si powder as reported by labs using ICP.

9.4.3.6 Arc Emission Spectroscopy

Table 9.4.3.6.1 Iron Concentration (wt. %) in SNR Powder

Material: Silicon Nitride, Reference Powder

Property: Fe

Method: 7

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
7	1	0.030	0.030	0.030	0.000
23	1	0.020	0.020	0.020	0.000
Means	2	0.020	0.030	0.025	0.007

Table 9.4.3.6.2 Iron Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder

Property: Fe

Method: 7

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
7	1	0.004	0.004	0.004	0.000
Means	1	0.004	0.004	0.004	0.000

Table 9.4.3.6.3 Iron Concentration (wt. %) in Si Powder

Material: Silicon

Property: Fe

Method: 7

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	1	0.030	0.030	0.030	0.000
Means	1	0.030	0.030	0.030	0.000

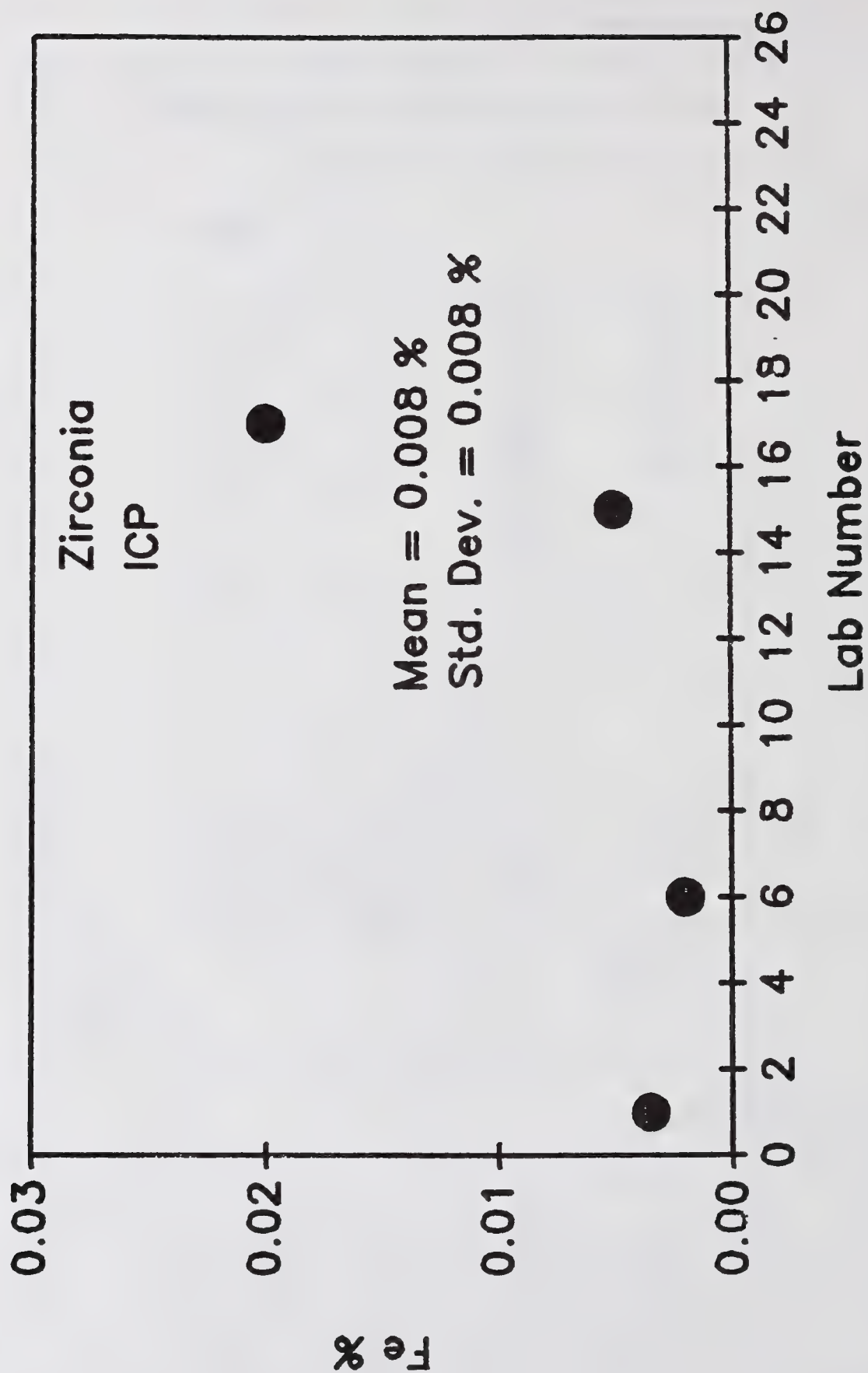


Figure 101. Iron concentration (wt. %) in YSZ powder as reported by labs using ICP.

9.4.3.7 DC Plasma - Emission Spectroscopy

Table 9.4.3.7.1 Iron Concentration (wt. %) in SNR Powder

Material: Silicon Nitride, Reference Powder (Figure 102)

Property: Fe

Method: 17

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
3	1	0.040	0.040	0.040	0.000
14	1	0.022	0.022	0.022	0.000
17	2	0.020	0.020	0.020	0.000
25	1	0.023	0.023	0.023	0.000
Means	4	0.020	0.040	0.026	0.009

Table 9.4.3.7.2 Iron Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder

Property: Fe

Method: 17

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
14	1	0.003	0.003	0.003	0.000
25	4	0.004	0.004	0.004	0.000
Means	2	0.003	0.004	0.004	0.001

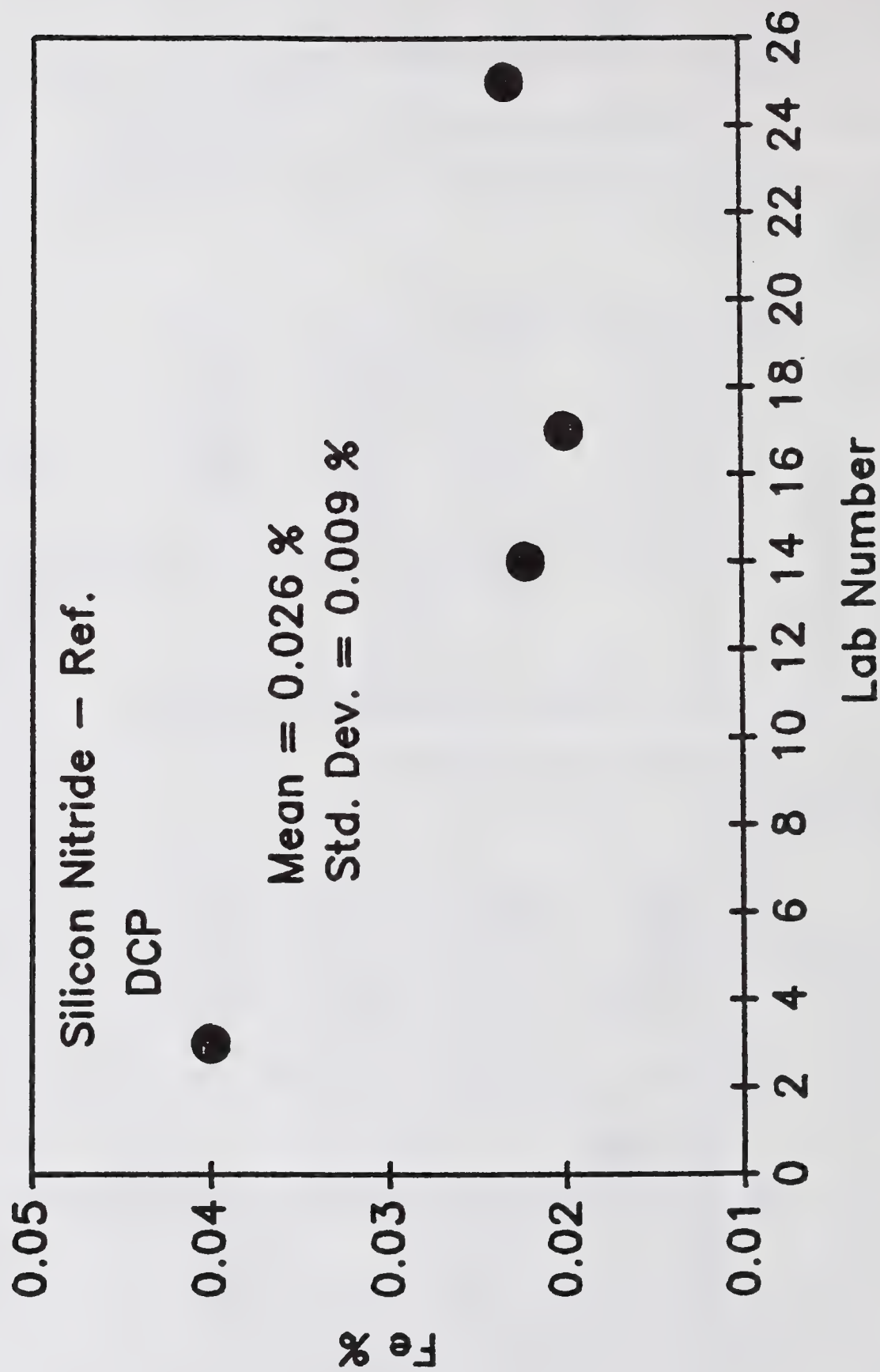


Figure 102. Iron concentration (wt. %) in SNR powder as reported by labs using DCP.

Table 9.4.3.7.3 Iron Concentration (wt. %) in SiC Powder

Material: Silicon Carbide

Property: Fe

Method: 17

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
25	6	0.002	0.003	0.003	0.001
Means	1	0.003	0.003	0.003	0.000

Table 9.4.3.7.4 Iron Concentration (wt. %) in Si Powder

Material: Silicon

Property: Fe

Method: 17

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
3	4	0.043	0.063	0.050	0.009
Means	1	0.050	0.050	0.050	0.000

Table 9.4.3.7.5 Iron Concentration (wt. %) in YSZ Powder

Material: Zirconia

Property: Fe

Method: 17

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
14	1	0.003	0.003	0.003	0.000
17	1	0.030	0.030	0.030	0.000
Means	2	0.003	0.030	0.016	0.019

9.4.3.8 Method not specified

Table 9.4.3.8 Iron Concentration (wt. %) in SiC Powder

Material: Silicon Carbide
Property: Fe
Method: 21

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
18	1	0.190	0.190	0.190	0.000
Means	1	0.190	0.190	0.190	0.000

9.4.4 Silicon in Yttria-Zirconia

9.4.4.1 Atomic Absorption Spectroscopy

Table 9.4.4.1 Silicon Concentration (wt. %) in YSZ Powder

Material: Zirconia
Property: Si
Method: 1

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
18	3	0.026	0.033	0.030	0.004
19	8	0.010	0.010	0.010	0.001
Means	2	0.010	0.030	0.020	0.014

9.4.4.2 Inductively Coupled Plasma - Emission Spectroscopy

Table 9.4.4.2 Silicon Concentration (wt. %) in YSZ Powder

Material: Zirconia
Property: Si
Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	2	0.006	0.007	0.006	0.001
6	1	0.004	0.004	0.004	0.000
17	1	0.010	0.010	0.010	0.000
Means	3	0.004	0.010	0.007	0.003

9.4.4.3 DC Plasma - Emission Spectroscopy

Table 9.4.4.3 Silicon Concentration (wt. %) in YSZ Powder

Material: Zirconia
Property: Si
Method: 17

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
17	1	0.010	0.010	0.010	0.000
Means	1	0.010	0.010	0.010	0.000

9.4.5 Titanium in Yttria-Zirconia

9.4.5.1 Neutron Activation Analysis

Table 9.4.5.1 Titanium Concentration (wt. %) in YSZ Powder

Material: Zirconia

Property: Ti

Method: 6

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
2	1	0.026	0.026	0.026	0.000
Means	1	0.026	0.026	0.026	0.000

9.4.5.2 X-ray Fluorescence Spectrometry

Table 9.4.5.2 Titanium Concentration (wt. %) in YSZ Powder

Material: Zirconia

Property: Ti

Method: 12

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
24	3	0.025	0.026	0.025	0.001
Means	1	0.025	0.025	0.025	0.000

9.4.5.3 Atomic Absorption Spectroscopy

Table 9.4.5.3 Titanium Concentration (wt. %) in YSZ Powder

Material: Zirconia
Property: Ti
Method: 1

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
14	2	0.024	0.025	0.024	0.001
19	8	0.040	0.060	0.051	0.008
Means	2	0.024	0.051	0.038	0.019

9.4.5.4 Inductively Coupled Plasma - Emission Spectroscopy

Table 9.4.5.4 Titanium Concentration (wt. %) in YSZ Powder

Material: Zirconia
Property: Ti
Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	2	0.026	0.028	0.027	0.001
6	1	0.018	0.018	0.018	0.000
15	1	0.024	0.024	0.024	0.000
Means	3	0.018	0.027	0.023	0.004

9.4.5.5 DC Plasma - Emission Spectroscopy

Table 9.4.5.5 Titanium Concentration (wt. %) in YSZ Powder

Material: Zirconia
Property: Ti
Method: 17

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
14	1	0.025	0.025	0.025	0.000
Means	1	0.025	0.025	0.025	0.000

9.4.6 Hafnium in Yttria-Zirconia

9.4.6.1 Mass Spectrometry

Table 9.4.6.1 Hafnium Concentration (wt. %) in YSZ Powder

Material: Zirconia
Property: Hf
Method: 5

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
8	3	0.60	0.60	0.60	0.00
Means	1	0.60	0.60	0.60	0.00

9.4.6.2 X-ray Fluorescence Spectrometry

Table 9.4.6.2 Hafnium Concentration (wt. %) in YSZ Powder

Material: Zirconia
Property: Hf
Method: 12

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
22	6	1.820	1.840	1.828	0.007
Means	1	1.828	1.828	1.828	0.000

9.4.6.3 Atomic Absorption Spectroscopy

Table 9.4.6.3 Hafnium Concentration (wt. %) in YSZ Powder

Material: Zirconia
Property: Hf
Method: 1

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
18	3	1.790	1.950	1.860	0.082
Means	1	1.860	1.860	1.860	0.000

9.4.6.4 Inductively Coupled Plasma - Emission Spectroscopy

Table 9.4.6.4 Hafnium Concentration (wt. %) in YSZ Powder

Material: Zirconia
Property: Hf
Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	2	1.720	1.720	1.720	0.000
6	1	0.080	0.080	0.080	0.000
17	1	1.770	1.770	1.770	0.000
Means	3	0.080	1.770	1.190	0.961

9.4.6.5 DC Plasma - Emission Spectroscopy

Table 9.4.6.5 Hafnium Concentration (wt. %) in YSZ Powder

Material: Zirconia
Property: Hf
Method: 17

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
17	1	1.760	1.760	1.760	0.000
Means	1	1.760	1.760	1.760	0.000

9.4.7 Tin in Yttria-Zirconia

9.4.7.1 Inductively Coupled Plasma - Emission Spectroscopy

Table 9.4.7.1 Tin Concentration (wt. %) in YSZ Powder

Material: Zirconia
Property: Sn
Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
6	2	0.014	0.059	0.036	0.032
Means	1	0.036	0.036	0.036	0.000

9.4.8 Neodymium in Yttria-Zirconia

9.4.8.1 Inductively Coupled Plasma - Emission Spectroscopy

Table 9.4.8.1 Neodymium Concentration (wt. %) in YSZ Powder

Material: Zirconia
Property: Nd
Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
6	1	0.028	0.028	0.028	0.00
Means	1	0.028	0.028	0.028	0.00

9.4.9 Holmium in Yttria-Zirconia

9.4.9.1 Inductively Coupled Plasma - Emission Spectroscopy

Table 9.4.9.1 Holmium Concentration (wt. %) in YSZ Powder

Material: Zirconia

Property: Ho

Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
6	1	0.025	0.025	0.025	0.000
Means	1	0.025	0.025	0.025	0.000

10. MINOR NONMETALLIC AND METALLIC IMPURITIES (Concentration < 100 ppm by weight) A. L. Dragoo, S. G. Malghan, S. M. Hsu and R. G. Munro, NIST

10.1. METHODS FOR MINOR NONMETALLIC AND METALLIC IMPURITIES

Methods used for determination of minor impurities have been described in Chapters 7, 8 and 9. Nearly all of the instrumental methods were used to measure one or more of the minor impurities. Impurities with relative concentrations in the range of 1 ppm to 100 ppm are termed "Minor Impurities."

Minor impurities are reported below for which determinations were included in this study. In most cases, the data were insufficient for an evaluation of the method. The impurities and the methods used are given without further comment unless four or more determinations were reported.

10.2. NONMETALLIC IMPURITIES (S, F, Cl) IN SILICON NITRIDE, SILICON CARBIDE AND SILICON

10.2.1. Sulfur

Table 10.2.1 Sulfur in Si_3N_4 , SiC and Si. Concentrations are in ppm by weight

Method	SNR	SNT	SiC	Si
MS	—	3	—	—
Combustion	110	—	—	—
ICP	182	—	44	5

— not available

10.2.2. Fluorine

Fluorine was reported at 40 ppm in silicon by one laboratory which used the specific ion electrode method and at 20 ppm by one laboratory which used potentiometric titration.

10.2.3. Chlorine

The results for chlorine determinations are summarized in Table 10.2.3. With the exception of the following three determinations, most results for a method and material were reported by only one laboratory and with less than four replications.

Lab 25 reported six determinations of the chlorine concentration in the SNT powder using ion chromatography: mean 87 ppm, s.d. 6 ppm.

Two laboratories reported the determination of chlorine concentration in SNT powder, total of six samples, using potentiometric titration: mean 87 ppm, s.d. 20 ppm.

Lab 5 reported four determinations of chlorine in Si by potentiometric titration: mean 40 ppm, s.d. 6 ppm.

Table 10.2.3 Chlorine in Si_3N_4 , SiC and Si. Concentrations are in ppm by weight

Method	SNR	SNT	SiC	Si
Ion Chromatography	35	87	241	210
NAA	—	80	—	—
SCA	55	68	—	—
Potentiometric Titration	—	87	51	40
Gravimetry	100	—	—	—

— not available

10.3. METALLIC IMPURITIES IN SILICON NITRIDE, SILICON CARBIDE AND SILICON (Na, Mg, K, Ti, Cr, Mn, Ni, W, B)

10.3.1. Sodium, Magnesium and Potassium

Results for Na and K impurities were, in most cases, for a single laboratory and a single determination. The average ICP result for Na in SiC was strongly skewed by a very large result obtained by one of the three laboratories reporting. If the outlying result is discarded, the average is about 18 ppm.

With the exception of the determination by mass spectrometry, Mg concentrations were replicated and were performed by more than one laboratory. The largest number of determinations by laboratories was that for Mg in the SNR powder by ICP. The means and standard deviations given in the Table 10.3.1 were averaged with respect to the laboratories. The ICP result for Mg in SNR powder is the average of the results of eight laboratories; replications by individual laboratories range from 1 to 11 determinations.

Table 10.3.1 Summary of Data for the Metallic Impurities Na, Mg and K in Silicon Nitride and Silicon Carbide for All Methods Reported. Concentration in ppm.

Material	Method	Na	Mg	K
Si ₃ N ₄ SNR	SCA	8	90(14)	—
	AAS	27	140(110)	—
	ICP	19	60(32)	—
	DCP	31	73(3)	—
	Grav.	100		100
Si ₃ N ₄ SNT	MS	3	1	2
	AAS	8	60(70)	21
	ICP	6	33(44)	—
	DCP	—	4	—
SiC	MS		10	—
	AAS	8	9	3
	ICP	1510	72	—
	DCP	—	3	—
Si	AAS	11	25(5)	37
	ICP	11	44(7)	—
	DCP	50	—	—

() replicate values; — not available

10.3.2. Titanium, Chromium and Manganese

The results for Ti, Cr and Mn impurities are summarized in Table 10.3.2. With the exception of determinations of Ti concentrations by ICP, the data were insufficient for any statistical evaluation. The large standard deviations which were obtained for the ICP determinations of Ti in the SNR and SNT powders suggest that sample preparation errors may have been a significant source of experimental error for the analyses of those powders.

Table 10.3.2 Summary of Data for the Metallic Impurities Ti, Cr and Mn in Silicon Nitride and Silicon Carbide for All Methods Reported. Concentration in ppm.

Material	Method	Ti	Cr	Mn
Si ₃ N ₄ SNR	OES	30	—	20
	AAS	15	—	6
	ICP	186(272)	3	7
	DCP	30	—	—
Si ₃ N ₄ SNT	NAA	—	6	>1
	MS	1	3	—
	AAS	—	—	3
	ICP	225	5	>1
SiC	XRF	60	—	—
	ICP	33(20)	23	>1
	DCP	38	—	—
Si	AAS	7	2	8(2)
	ICP	8(2)	4	8

() replicate values; — not available

10.3.3. Nickel, Tungsten and Boron

The results for Ni, W and B impurities are summarized in Table 10.3.3. The data were insufficient for statistical evaluation.

Table 10.3.3 Summary of Data for the Metallic Impurities Ni, W and B in Silicon Nitride and Silicon Carbide for All Methods Reported. Concentration in ppm.

Material	Method	Ni	W	B
Si ₃ N ₄ SNR	OES	—	—	10
	AAS	—	—	20
	ICP	4	—	—
	DCP	—	50	—
Si ₃ N ₄ SNT	MS	2	—	—
	OES	5	—	—
	ICP	5(1)	—	—
SiC	ICP	30	—	—
Si	AAS	—	—	10
Si	ICP	6(3)	—	—

— not available

10.4. METALLIC IMPURITIES IN YTTRIA-ZIRCONIA (Na, Mg, K, Ga, Ba)

The results are summarized in Table 10.4.1.

Table 10.4.1 Summary of Data for the Metallic Impurities Na, Mg, K, Ga and Ba in Yttria-Zirconia for All Methods Reported. Concentration in ppm.

Method	Na	Mg	K	Ga	Ba
NAA	77	—	6	—	—
MS	230	—	—	—	—
AAS	70(31)	53(45)	50	—	—
ICP	22	48(36)	—	58	330
DCP	70	20	—	—	—

— not available

10.5. SULFUR IN SILICON NITRIDE, SILICON CARBIDE AND SILICON

Sulfur is classed here as a minor impurity although it was detected as a less abundant major impurity, in the range of a few hundred ppm in some measurements. However, in other measurements and for the SNT powder especially, it was detected as a minor impurity.

10.5.1 Mass Spectroscopy

Table 10.5.1 Sulfur Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder
Property: S
Method: 5

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
8	2	0.0001	0.0005	0.0003	0.0003
Means	1	0.0003	0.0003	0.0003	0.0000

10.5.2 Combustion Analysis

Table 10.5.2 Sulfur Concentration (wt. %) in SNR Powder

Material: Silicon Nitride, Reference Powder

Property: S

Method: 8

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
17	1	0.011	0.011	0.011	0.000
Means	1	0.011	0.011	0.011	0.000

10.5.3 Inductively Coupled Plasma - Emission Spectroscopy

Table 10.5.3.1 Sulfur Concentration (wt. %) in SNR Powder

Material: Silicon Nitride, Reference Powder

Property: S

Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
6	2	0.0040	0.0323	0.0182	0.0200
Means	1	0.0182	0.0182	0.0182	0.0000

Table 10.5.3.2 Sulfur Concentration (wt. %) in SiC Powder

Material: Silicon Carbide

Property: S

Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
6	1	0.0044	0.0044	0.0044	0.0000
Means	1	0.0044	0.0044	0.0044	0.0000

Table 10.5.3.3 Sulfur Concentration (wt. %) in Si Powder

Material: Silicon

Property: S

Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
6	1	0.0005	0.0005	0.0005	0.0000
Means	1	0.0005	0.0005	0.0005	0.0000

10.6. FLUORINE IN SILICON10.6.1 Electrochemical Methods - Potentiometric Titration

Table 10.6.1 Fluorine Concentration (wt. %) in Si Powder

Material: Silicon

Property: F

Method: 13

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
5	4	0.002	0.002	0.002	0.000
Means	1	0.002	0.002	0.002	0.000

10.6.2 Electrochemical Methods - Specific Ion Electrode

Table 10.6.2 Fluorine Concentration (wt. %) in Si Powder

Material: Silicon

Property: F

Method: 4

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
6	1	0.004	0.004	0.004	0.000
Means	1	0.004	0.004	0.004	0.000

10.7. CHLORINE IN SILICON NITRIDE, SILICON CARBIDE AND SILICON

10.7.1 Neutron Activation Analysis

Table 10.7.1 Chlorine Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder

Property: Cl

Method: 6

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
2	1	0.008	0.008	0.008	0.000
Means	1	0.008	0.008	0.008	0.000

10.7.2 Arc Emission Spectroscopy

Table 10.7.2.1 Chlorine Concentration (wt. %) in SNR Powder

Material: Silicon Nitride, Reference Powder
Property: Cl
Method: 7

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
22	2	0.0046	0.0063	0.0055	0.0012
Means	1	0.0055	0.0055	0.0055	0.0000

Table 10.7.2.2 Chlorine Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder
Property: Cl
Method: 7

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
22	3	0.0056	0.0075	0.0068	0.0011
Means	1	0.0068	0.0068	0.0068	0.0000

10.7.3 Ion Chromatography

Table 10.7.3.1 Chlorine Concentration (wt. %) in SNR Powder

Material: Silicon Nitride, Reference Powder
Property: Cl
Method: 3

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
6	2	0.0004	0.0065	0.0034	0.0043
Means	1	0.0034	0.0034	0.0034	0.0000

Table 10.7.3.2 Chlorine Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder

Property: Cl

Method: 3

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
25	6	0.0082	0.0094	0.0087	0.0006
Means	1	0.0087	0.0087	0.0087	0.0000

Table 10.7.3.3 Chlorine Concentration (wt. %) in SiC Powder

Material: Silicon Carbide

Property: Cl

Method: 3

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
6	1	0.0241	0.0241	0.0241	0.0000
Means	1	0.0241	0.0241	0.0241	0.0000

Table 10.7.3.4 Chlorine Concentration (wt. %) in Si Powder

Material: Silicon

Property: Cl

Method: 3

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
6	1	0.021	0.021	0.021	0.000
Means	1	0.021	0.021	0.021	0.000

10.7.4 Electrochemical Methods - Potentiometric Titration

Table 10.7.4.1 Chlorine Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder
Property: Cl
Method: 13

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
5	4	0.0069	0.0076	0.0072	0.0003
21	2	0.0100	0.0102	0.0101	0.0001
Means	2	0.0072	0.0101	0.0087	0.0020

Table 10.7.4.2 Chlorine Concentration (wt. %) in SiC Powder

Material: Silicon Carbide
Property: Cl
Method: 13

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
21	3	0.0049	0.0055	0.0051	0.0003
Means	1	0.0051	0.0051	0.0051	0.0000

Table 10.7.4.3 Chlorine Concentration (wt. %) in Si Powder

Material: Silicon
Property: Cl
Method: 13

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
5	4	0.0033	0.0046	0.0040	0.0006
Means	1	0.0040	0.0040	0.0040	0.0000

10.7.5 Gravimetry

Table 10.7.5 Chlorine Concentration (wt. %) in SNR Powder

Material: Silicon Nitride, Reference Powder
Property: Cl
Method: 20

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
24	1	0.010	0.010	0.010	0.000
Means	1	0.010	0.010	0.010	0.000

10.8. SODIUM IN ALL POWDERS

10.8.1 Mass Spectrometry

Table 10.8.1.1 Sodium Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder
Property: Na
Method: 5

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
8	1	0.0003	0.0003	0.0003	0.0000
Means	1	0.0003	0.0003	0.0003	0.0000

Table 10.8.1.2 Sodium Concentration (wt. %) in YSZ Powder

Material: Zirconia

Property: Na

Method: 5

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
8	3	0.0100	0.0500	0.0233	0.0231
Means	1	0.0233	0.0233	0.0233	0.0000

10.8.2 Neutron Activation Analysis

Table 10.8.2 Sodium Concentration (wt. %) in YSZ Powder

Material: Zirconia

Property: Na

Method: 6

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
2	1	0.0077	0.0077	0.0077	0.0000
Means	1	0.0077	0.0077	0.0077	0.0000

10.8.3 Atomic Absorption Spectroscopy

Table 10.8.3.1 Sodium Concentration (wt. %) in SNR Powder

Material: Silicon Nitride, Reference Powder

Property: Na

Method: 1

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
14	2	0.0021	0.0024	0.0022	0.0002
16	6	0.0010	0.0070	0.0028	0.0023
Means	2	0.0022	0.0028	0.0025	0.0004

Table 10.8.3.2 Sodium Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder

Property: Na

Method: 1

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
14	2	0.008	0.008	0.008	0.000
Means	1	0.008	0.008	0.008	0.000

Table 10.8.3.3 Sodium Concentration (wt. %) in SiC Powder

Material: Silicon Carbide

Property: Na

Method: 1

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
16	1	0.0008	0.0008	0.0008	0.0000
Means	1	0.0008	0.0008	0.0008	0.0000

Table 10.8.3.4 Sodium Concentration (wt. %) in Si Powder

Material: Silicon

Property: Na

Method: 1

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	1	0.0010	0.0010	0.0010	0.0000
16	1	0.0012	0.0012	0.0012	0.0000
Means	2	0.0010	0.0012	0.0011	0.0001

Table 10.8.3.5 Sodium Concentration (wt. %) in YSZ Powder

Material: Zirconia

Property: Na

Method: 1

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
14	2	0.0036	0.0039	0.0037	0.0002
19	8	0.0100	0.0100	0.0100	0.0000
24	3	0.0070	0.0080	0.0073	0.0006
Means	3	0.0038	0.0100	0.0070	0.0031

10.8.4 Inductively Coupled Plasma - Emission Spectroscopy

Table 10.8.4.1 Sodium Concentration (wt. %) in SNR Powder

Material: Silicon Nitride, Reference Powder

Property: Na

Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
6	1	0.0047	0.0047	0.0047	0.0000
13	2	0.0300	0.0600	0.0450	0.0212
15	1	0.0012	0.0012	0.0012	0.0000
20	1	0.0002	0.0002	0.0002	0.0000
Means	4	0.0002	0.0450	0.0128	0.0216

Table 10.8.4.2 Sodium Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder

Property: Na

Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
6	1	0.0119	0.0119	0.0119	0.0000
20	1	0.0002	0.0002	0.0002	0.0000
Means	2	0.0002	0.0119	0.0061	0.0083

Table 10.8.4.3 Sodium Concentration (wt. %) in SiC Powder

Material: Silicon Carbide

Property: Na

Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
6	1	0.0011	0.0011	0.0011	0.0000
15	1	0.0024	0.0024	0.0024	0.0000
20	2	0.3000	0.6000	0.4500	0.2121
Means	3	0.0011	0.4500	0.1512	0.2588

Table 10.8.4.4 Sodium Concentration (wt. %) in Si Powder

Material: Silicon

Property: Na

Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
15	1	0.0012	0.0012	0.0012	0.0000
20	2	0.0008	0.0013	0.0010	0.0004
Means	2	0.0011	0.0012	0.0011	0.0001

Table 10.8.4.5 Sodium Concentration (wt. %) in YSZ Powder

Material: Zirconia

Property: Na

Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
15	1	0.0022	0.0022	0.0022	0.0000
Means	1	0.0022	0.0022	0.0022	0.0000

10.8.5 Arc Emission Spectroscopy

Table 10.8.5.1 Sodium Concentration (wt. %) in SNR Powder

Material: Silicon Nitride, Reference Powder

Property: Na

Method: 7

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
7	1	0.0008	0.0008	0.0008	0.0000
Means	1	0.0008	0.0008	0.0008	0.0000

10.8.6 DC Plasma - Emission Spectroscopy

Table 10.8.6.1 Sodium Concentration (wt. %) in SNR Powder

Material: Silicon Nitride, Reference Powder

Property: Na

Method: 17

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
14	1	0.0031	0.0031	0.0031	0.0000
Means	1	0.0031	0.0031	0.0031	0.0000

Table 10.8.6.2 Sodium Concentration (wt. %) in YSZ Powder

Material: Zirconia
 Property: Na
 Method: 17

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
14	1	0.0070	0.0070	0.0070	0.0000
Means	1	0.0070	0.0070	0.0070	0.0000

10.8.7 Gravimetry

Table 10.8.7.1 Sodium Concentration (wt. %) in SNR Powder

Material: Silicon Nitride, Reference Powder
 Property: Na
 Method: 20

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
24	1	0.0100	0.0100	0.0100	0.0000
Means	1	0.0100	0.0100	0.0100	0.0000

10.9. MAGNESIUM IN ALL POWDERS

10.9.1 Mass Spectroscopy

Table 10.9.1.1 Magnesium Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder
Property: Mg
Method: 5

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
8	2	0.0001	0.0001	0.0001	0.0000
Means	1	0.0001	0.0001	0.0001	0.0000

Table 10.9.1.2 Magnesium Concentration (wt. %) in SiC Powder

Material: Silicon Carbide
Property: Mg
Method: 5

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
8	1	0.001	0.001	0.001	0.000
Means	1	0.001	0.001	0.001	0.000

Table 10.9.1.3 Magnesium Concentration (wt. %) in Si Powder

Material: Silicon
Property: Mg
Method: 5

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
8	1	0.003	0.003	0.003	0.000
Means	1	0.003	0.003	0.003	0.000

10.9.2 Atomic Absorption Spectroscopy

Table 10.9.2.1 Magnesium Concentration (wt. %) in SNR Powder

Material: Silicon Nitride, Reference Powder

Property: Mg

Method: 1

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
14	2	0.0080	0.0082	0.0081	0.0001
16	6	0.0060	0.0070	0.0065	0.0005
24	3	0.0200	0.0360	0.0267	0.0083
Means	3	0.0065	0.0267	0.0138	0.0112

Table 10.9.2.2 Magnesium Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder

Property: Mg

Method: 1

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
14	2	0.0010	0.0010	0.0010	0.0000
24	2	0.0090	0.0130	0.0110	0.0028
Means	2	0.0010	0.0110	0.0060	0.0071

Table 10.9.2.3 Magnesium Concentration (wt. %) in SiC Powder

Material: Silicon Carbide

Property: Mg

Method: 1

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
10	1	0.0016	0.0016	0.0016	0.0000
16	2	0.0002	0.0002	0.0002	0.0000
Means	2	0.0002	0.0016	0.0009	0.0010

Table 10.9.2.4 Magnesium Concentration (wt. %) in Si Powder

Material: Silicon

Property: Mg

Method: 1

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	1	0.0020	0.0020	0.0020	0.0000
16	2	0.0030	0.0030	0.0030	0.0000
19	9	0.0020	0.0032	0.0026	0.0004
Means	3	0.0020	0.0030	0.0025	0.0005

Table 10.9.2.5 Magnesium Concentration (wt. %) in YSZ Powder

Material: Zirconia

Property: Mg

Method: 1

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
14	2	0.0009	0.0011	0.0010	0.0001
19	8	0.0100	0.0100	0.0100	0.0000
24	3	0.0050	0.0050	0.0050	0.0000
Means	3	0.0010	0.0100	0.0053	0.0045

10.9.3 Inductively Coupled Plasma - Emission Spectroscopy

Table 10.9.3.1 Magnesium Concentration (wt. %) in SNR Powder

Material: Silicon Nitride, Reference Powder
Property: Mg
Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	2	0.0060	0.0060	0.0060	0.0000
5	1	0.0063	0.0063	0.0063	0.0000
6	1	0.0065	0.0065	0.0065	0.0000
7	6	0.0073	0.0080	0.0076	0.0003
13	11	0.0100	0.0200	0.0109	0.0030
15	1	0.0071	0.0071	0.0071	0.0000
20	1	0.0002	0.0002	0.0002	0.0000
22	4	0.0030	0.0040	0.0033	0.0005
Means	8	0.0002	0.0109	0.0060	0.0032

Table 10.9.3.2 Magnesium Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder
Property: Mg
Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
15	1	0.0002	0.0002	0.0002	0.0000
17	1	0.0083	0.0083	0.0083	0.0000
20	1	0.0014	0.0014	0.0014	0.0000
Means	3	0.0002	0.0083	0.0033	0.0044

Table 10.9.3.3 Magnesium Concentration (wt. %) in SiC Powder

Material: Silicon Carbide

Property: Mg

Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
15	1	0.0003	0.0003	0.0003	0.0000
20	2	0.0110	0.0170	0.0140	0.0042
Means	2	0.0003	0.0140	0.0072	0.0097

Table 10.9.3.4 Magnesium Concentration (wt. %) in Si Powder

Material: Silicon

Property: Mg

Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
6	1	0.0042	0.0042	0.0042	0.0000
15	1	0.0037	0.0037	0.0037	0.0000
20	2	0.0029	0.0074	0.0052	0.0032
Means	3	0.0037	0.0052	0.0044	0.0007

Table 10.9.3.5 Magnesium Concentration (wt. %) in YSZ Powder

Material: Zirconia

Property: Mg

Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	2	0.0010	0.0010	0.0010	0.0000
6	1	0.0093	0.0093	0.0093	0.0000
15	1	0.0060	0.0060	0.0060	0.0000
17	1	0.0030	0.0030	0.0030	0.0000
Means	4	0.0010	0.0093	0.0048	0.0036

10.9.4 Arc Emission Spectroscopy

Table 10.9.4 Magnesium Concentration (wt. %) in SNR Powder

Material: Silicon Nitride, Reference Powder

Property: Mg

Method: 7

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
7	1	0.0080	0.0080	0.0080	0.0000
23	1	0.0100	0.0100	0.0100	0.0000
Means	2	0.0080	0.0100	0.0090	0.0014

10.9.5 DC Plasma - Emission Spectroscopy

Table 10.9.5.1 Magnesium Concentration (wt. %) in SNR Powder

Material: Silicon Nitride, Reference Powder

Property: Mg

Method: 17

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
14	1	0.0074	0.0074	0.0074	0.0000
17	2	0.0070	0.0070	0.0070	0.0000
25	1	0.0075	0.0075	0.0075	0.0000
Means	3	0.0070	0.0075	0.0073	0.0003

Table 10.9.5.2 Magnesium Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder

Property: Mg

Method: 17

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
14	1	0.0004	0.0004	0.0004	0.0000
Means	1	0.0004	0.0004	0.0004	0.0000

Table 10.9.5.3 Magnesium Concentration (wt. %) in SiC Powder

Material: Silicon Carbide

Property: Mg

Method: 17

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
25	4	0.0001	0.0004	0.0003	0.0001
Means	1	0.0003	0.0003	0.0003	0.0000

Table 10.9.5.4 Magnesium Concentration (wt. %) in Si Powder

Material: Silicon

Property: Mg

Method: 17

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
3	4	0.0050	0.0050	0.0050	0.0000
Means	1	0.0050	0.0050	0.0050	0.0000

Table 10.9.5.5 Magnesium Concentration (wt. %) in YSZ Powder

Material: Zirconia
Property: Mg
Method: 17

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
17	1	0.0020	0.0020	0.0020	0.0000
Means	1	0.0020	0.0020	0.0020	0.0000

10.10. POTASSIUM IN ALL POWDERS

10.10.1 Mass Spectroscopy

Table 10.10.1 Potassium Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder
Property: K
Method: 5

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
8	3	0.0002	0.0002	0.0002	0.0000
Means	1	0.0002	0.0002	0.0002	0.0000

10.10.2 Neutron Activation Analysis

Table 10.10.2 Potassium Concentration (wt. %) in YSZ Powder

Material: Zirconia
Property: K
Method: 6

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
2	1	0.0006	0.0006	0.0006	0.0000
Means	1	0.0006	0.0006	0.0006	0.0000

10.10.3 Atomic Absorption Spectroscopy

Table 10.10.3.1 Potassium Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder
Property: K
Method: 1

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
14	2	0.0020	0.0022	0.0021	0.0001
Means	1	0.0021	0.0021	0.0021	0.0000

Table 10.10.3.2 Potassium Concentration (wt. %) in SiC Powder

Material: Silicon Carbide
Property: K
Method: 1

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
16	1	0.0003	0.0003	0.0003	0.0000
Means	1	0.0003	0.0003	0.0003	0.0000

Table 10.10.3.3 Potassium Concentration (wt. %) in Si Powder

Material: Silicon
Property: K
Method: 1

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	1	0.0100	0.0100	0.0100	0.0000
16	2	0.0005	0.0007	0.0006	0.0001
Means	2	0.0006	0.0100	0.0053	0.0066

Table 10.10.3.4 Potassium Concentration (wt. %) in YSZ Powder

Material: Zirconia
Property: K
Method: 1

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
24	3	0.0050	0.0050	0.0050	0.0000
Means	1	0.0050	0.0050	0.0050	0.0000

10.10.4 Gravimetry

Table 10.10.4 Potassium Concentration (wt. %) in SNR Powder

Material: Silicon Nitride, Reference Powder
Property: K
Method: 20

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
24	1	0.0100	0.0100	0.0100	0.0000
Means	1	0.0100	0.0100	0.0100	0.0000

10.11. TITANIUM IN SILICON NITRIDE, SILICON CARBIDE AND SILICON

10.11.1 Mass Spectrometry

Table 10.11.1 Titanium Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder

Property: Ti

Method: 5

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
8	1	0.0001	0.0001	0.0001	0.0000
Means	1	0.0001	0.0001	0.0001	0.0000

10.11.2 X-ray Fluorescence Spectrometry

Table 10.11.2 Titanium Concentration (wt. %) in SiC Powder

Material: Silicon Carbide

Property: Ti

Method: 12

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
10	1	0.0060	0.0060	0.0060	0.0000
Means	1	0.0060	0.0060	0.0060	0.0000

10.11.3 Atomic Absorption Spectroscopy

Table 10.11.3.1 Titanium Concentration (wt. %) in SNR Powder

Material: Silicon Nitride, Reference Powder

Property: Ti

Method: 1

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
14	1	0.0015	0.0015	0.0015	0.0000
Means	1	0.0015	0.0015	0.0015	0.0000

Table 10.11.3.2 Titanium Concentration (wt. %) in Si Powder

Material: Silicon

Property: Ti

Method: 1

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
19	9	0.0006	0.0008	0.0007	0.0001
Means	1	0.0007	0.0007	0.0007	0.0000

10.11.4 Inductively Coupled Plasma - Emission Spectroscopy

Table 10.11.4.1 Titanium Concentration (wt. %) in SNR Powder

Material: Silicon Nitride, Reference Powder

Property: Ti

Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
7	6	0.0028	0.0030	0.0029	0.0001
15	1	0.0030	0.0030	0.0030	0.0000
18	1	0.0500	0.0500	0.0500	0.0000
Means	3	0.0029	0.0500	0.0186	0.0272

Table 10.11.4.2 Titanium Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder

Property: Ti

Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
17	1	0.0149	0.0149	0.0149	0.0000
18	1	0.0300	0.0300	0.0300	0.0000
Means	2	0.0149	0.0300	0.0224	0.0107

Table 10.11.4.3 Titanium Concentration (wt. %) in SiC Powder

Material: Silicon Carbide

Property: Ti

Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
6	1	0.0047	0.0047	0.0047	0.0000
15	1	0.0043	0.0043	0.0043	0.0000
20	2	0.0010	0.0010	0.0010	0.0000
Means	3	0.0010	0.0047	0.0033	0.0020

Table 10.11.4.4 Titanium Concentration (wt. %) in Si Powder

Material: Silicon
Property: Ti
Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
7	2	0.0010	0.0010	0.0010	0.0000
15	1	0.0007	0.0007	0.0007	0.0000
20	2	0.0007	0.0008	0.0008	0.0001
Means	3	0.0007	0.0010	0.0008	0.0002

10.11.5 Arc Emission Spectroscopy

Table 10.11.5 Titanium Concentration (wt. %) in SNR Powder

Material: Silicon Nitride, Reference Powder
Property: Ti
Method: 7

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
7	1	0.0030	0.0030	0.0030	0.0000
Means	1	0.0030	0.0030	0.0030	0.0000

10.11.6 DC Plasma - Emission Spectroscopy

Table 10.11.6.1 Titanium Concentration (wt. %) in SNR Powder

Material: Silicon Nitride, Reference Powder

Property: Ti

Method: 17

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
25	1	0.0030	0.0030	0.0030	0.0000
Means	1	0.0030	0.0030	0.0030	0.0000

Table 10.11.6.2 Titanium Concentration (wt. %) in SiC Powder

Material: Silicon Carbide

Property: Ti

Method: 17

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
25	6	0.0032	0.0044	0.0038	0.0004
Means	1	0.0038	0.0038	0.0038	0.0000

10.12. CHROMIUM IN SILICON NITRIDE, SILICON CARBIDE AND SILICON

10.12.1 Mass Spectroscopy

Table 10.12.1 Chromium Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder
Property: Cr
Method: 5

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
8	3	0.0002	0.0003	0.0003	0.0001
Means	1	0.0003	0.0003	0.0003	0.0000

10.12.2 Neutron Activation Analysis

Table 10.12.2 Chromium Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder
Property: Cr
Method: 6

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
2	1	0.0006	0.0006	0.0006	0.0000
Means	1	0.0006	0.0006	0.0006	0.0000

10.12.3 Atomic Absorption Spectroscopy

Table 10.12.3 Chromium Concentration (wt. %) in Si Powder

Material: Silicon
Property: Cr
Method: 1

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
19	9	0.0001	0.0003	0.0002	0.0001
Means	1	0.0002	0.0002	0.0002	0.0000

10.12.4 Inductively Coupled Plasma - Emission Spectroscopy

Table 10.12.4.1 Chromium Concentration (wt. %) in SNR Powder

Material: Silicon Nitride, Reference Powder
Property: Cr
Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
15	1	0.0003	0.0003	0.0003	0.0000
Means	1	0.0003	0.0003	0.0003	0.0000

Table 10.12.4.2 Chromium Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder
Property: Cr
Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
15	1	0.0005	0.0005	0.0005	0.0000
Means	1	0.0005	0.0005	0.0005	0.0000

Table 10.12.4.3 Chromium Concentration (wt. %) in SiC Powder

Material: Silicon Carbide
Property: Cr
Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
15	1	0.0023	0.0023	0.0023	0.0000
Means	1	0.0023	0.0023	0.0023	0.0000

Table 10.12.4.4 Chromium Concentration (wt. %) in Si Powder

Material: Silicon
Property: Cr
Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
15	1	0.0004	0.0004	0.0004	0.0000
Means	1	0.0004	0.0004	0.0004	0.0000

10.13. MANGANESE IN SILICON NITRIDE, SILICON CARBIDE AND SILICON

10.13.1 Neutron Activation Analysis

Table 10.13.1 Manganese Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder

Property: Mn

Method: 6

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
2	1	0.0000	0.0000	0.0000	0.0000
Means	1	0.0000	0.0000	0.0000	0.0000

10.13.2 Atomic Absorption Spectroscopy

Table 10.13.2.1 Manganese Concentration (wt. %) in SNR Powder

Material: Silicon Nitride, Reference Powder

Property: Mn

Method: 1

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
16	6	0.0005	0.0008	0.0006	0.0001
Means	1	0.0006	0.0006	0.0006	0.0000

Table 10.13.2.2 Manganese Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder

Property: Mn

Method: 1

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
16	1	0.0003	0.0003	0.0003	0.0000
Means	1	0.0003	0.0003	0.0003	0.0000

Table 10.13.2.3 Manganese Concentration (wt. %) in Si Powder

Material: Silicon

Property: Mn

Method: 1

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	1	0.0010	0.0010	0.0010	0.0000
16	2	0.0007	0.0007	0.0007	0.0000
19	9	0.0007	0.0008	0.0007	0.0000
Means	3	0.0007	0.0010	0.0008	0.0002

10.13.3 Inductively Coupled Plasma - Emission Spectroscopy

Table 10.13.3.1 Manganese Concentration (wt. %) in SNR Powder

Material: Silicon Nitride, Reference Powder

Property: Mn

Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
15	1	0.0007	0.0007	0.0007	0.0000
19	1	0.0006	0.0006	0.0006	0.0000
Means	2	0.0006	0.0007	0.0006	0.0001

Table 10.13.3.2 Manganese Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder
 Property: Mn
 Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
15	1	0.0000	0.0000	0.0000	0.0000
19	7	0.0000	0.0001	0.0000	0.0001
Means	2	0.0000	0.0000	0.0000	0.0000

Table 10.13.3.3 Manganese Concentration (wt. %) in SiC Powder

Material: Silicon Carbide
 Property: Mn
 Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
15	1	0.0001	0.0001	0.0001	0.0000
Means	1	0.0001	0.0001	0.0001	0.0000

Table 10.13.3.4 Manganese Concentration (wt. %) in Si Powder

Material: Silicon
 Property: Mn
 Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
7	2	0.0010	0.0010	0.0010	0.0000
15	1	0.0006	0.0006	0.0006	0.0000
Means	2	0.0006	0.0010	0.0008	0.0003

10.13.4 Arc Emission Spectroscopy

Table 10.13.4 Manganese Concentration (wt. %) in SNR Powder

Material: Silicon Nitride, Reference Powder
Property: Mn
Method: 7

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
7	1	0.0020	0.0020	0.0020	0.0000
Means	1	0.0020	0.0020	0.0020	0.0000

10.14. NICKEL IN SILICON NITRIDE, SILICON CARBIDE AND SILICON

10.14.1 Mass Spectrometry

Table 10.14.1 Nickel Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder
Property: Ni
Method: 5

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
8	3	0.0001	0.0004	0.0002	0.0002
Means	1	0.0002	0.0002	0.0002	0.0000

10.14.2 Atomic Absorption Spectroscopy

Table 10.14.2.1 Nickel Concentration (wt. %) in SNR Powder

Material: Silicon Nitride, Reference Powder
Property: Ni
Method: 1

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
24	1	0.0100	0.0100	0.0100	0.0000
Means	1	0.0100	0.0100	0.0100	0.0000

Table 10.14.2.2 Nickel Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder
Property: Ni
Method: 1

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
24	1	0.0280	0.0280	0.0280	0.0000
Means	1	0.0280	0.0280	0.0280	0.0000

Table 10.14.2.3 Nickel Concentration (wt. %) in Si Powder

Material: Silicon
Property: Ni
Method: 1

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	1	0.0010	0.0010	0.0010	0.0000
19	9	0.0002	0.0005	0.0003	0.0001
Means	2	0.0003	0.0010	0.0007	0.0005

10.14.3 Inductively Coupled Plasma - Emission Spectroscopy

Table 10.14.3.1 Nickel Concentration (wt. %) in SNR Powder

Material: Silicon Nitride, Reference Powder
Property: Ni
Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
19	1	0.0003	0.0003	0.0003	0.0000
20	1	0.0005	0.0005	0.0005	0.0000
Means	2	0.0003	0.0005	0.0004	0.0001

Table 10.14.3.2 Nickel Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder
Property: Ni
Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
15	1	0.0004	0.0004	0.0004	0.0000
19	7	0.0005	0.0006	0.0006	0.0001
20	1	0.0005	0.0005	0.0005	0.0000
Means	3	0.0004	0.0006	0.0005	0.0001

Table 10.14.3.3 Nickel Concentration (wt. %) in SiC Powder

Material: Silicon Carbide
 Property: Ni
 Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
15	1	0.0005	0.0005	0.0005	0.0000
20	2	0.0050	0.0050	0.0050	0.0000
Means	2	0.0005	0.0050	0.0027	0.0032

Table 10.14.3.4 Nickel Concentration (wt. %) in Si Powder

Material: Silicon
 Property: Ni
 Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
7	1	0.0010	0.0010	0.0010	0.0000
15	1	0.0004	0.0004	0.0004	0.0000
20	2	0.0005	0.0005	0.0005	0.0000
Means	3	0.0004	0.0010	0.0006	0.0003

10.14.4 Arc Emission Spectroscopy

Table 10.14.4 Nickel Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder
 Property: Ni
 Method: 7

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
7	1	0.0005	0.0005	0.0005	0.0000
Means	1	0.0005	0.0005	0.0005	0.0000

10.15. TUNGSTEN IN SILICON NITRIDE

10.15.1 DC Plasma - Emission Spectroscopy

Table 10.15.1 Tungsten Concentration (wt. %) in SNR Powder

Material: Silicon Nitride, Reference Powder

Property: W

Method: 17

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
17	1	0.0050	0.0050	0.0050	0.0000
Means	1	0.0050	0.0050	0.0050	0.0000

10.16. BORON IN SILICON NITRIDE, SILICON CARBIDE AND SILICON

10.16.1 Atomic Absorption Spectroscopy

Table 10.16.1.1 Boron Concentration (wt. %) in SNR Powder

Material: Silicon Nitride, Reference Powder

Property: B

Method: 1

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
23	1	0.0020	0.0020	0.0020	0.0000
Means	1	0.0020	0.0020	0.0020	0.0000

Table 10.16.1.2 Boron Concentration (wt. %) in Si Powder

Material: Silicon

Property: B

Method: 1

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	1	0.0010	0.0010	0.0010	0.0000
Means	1	0.0010	0.0010	0.0010	0.0000

10.16.2 Inductively Coupled Plasma - Emission Spectroscopy

Table 10.16.2.1 Boron Concentration (wt. %) in SNR Powder

Material: Silicon Nitride, Reference Powder

Property: B

Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
1	2	0.0100	0.0200	0.0150	0.0071
15	1	0.0004	0.0004	0.0004	0.0000
Means	2	0.0004	0.0150	0.0077	0.0103

Table 10.16.2.2 Boron Concentration (wt. %) in SNT Powder

Material: Silicon Nitride, Test Powder

Property: B

Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
15	1	0.0002	0.0002	0.0002	0.0000
Means	1	0.0002	0.0002	0.0002	0.0000

Table 10.16.2.3 Boron Concentration (wt. %) in SiC Powder

Material: Silicon Carbide
 Property: B
 Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
15	1	0.0005	0.0005	0.0005	0.0000
21	3	0.0005	0.0006	0.0006	0.0001
Means	2	0.0005	0.0006	0.0005	0.0000

Table 10.16.2.4 Boron Concentration (wt. %) in Si Powder

Material: Silicon
 Property: B
 Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
15	1	0.0014	0.0014	0.0014	0.0000
Means	1	0.0014	0.0014	0.0014	0.0000

10.16.3 Arc Emission Spectroscopy

Table 10.16.2 Boron Concentration (wt. %) in SNR Powder

Material: Silicon Nitride, Reference Powder
 Property: B
 Method: 7

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
7	1	0.0010	0.0010	0.0010	0.0000
Means	1	0.0010	0.0010	0.0010	0.0000

10.17. GALLIUM IN YTTRIA-ZIRCONIA

10.17.1 Inductively Coupled Plasma - Emission Spectroscopy

Table 10.17.1 Gallium Concentration (wt. %) in YSZ Powder

Material: Zirconia
Property: Ga
Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
6	1	0.0058	0.0058	0.0058	0.0000
Means	1	0.0058	0.0058	0.0058	0.0000

10.18. BARIUM IN YTTRIA-ZIRCONIA

10.18.1 Inductively Coupled Plasma - Emission Spectroscopy

Table 10.18.2 Barium Concentration (wt. %) in YSZ Powder

Material: Zirconia
Property: Ba
Method: 2

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
6	1	0.0330	0.0330	0.0330	0.0000
Means	1	0.0330	0.0330	0.0330	0.0000

10.19. STATISTICAL ANALYSIS, R. Munro, NIST

Atomic absorption spectroscopy and inductively coupled plasma emission spectroscopy were used most often to determine the amount of metallic impurities in the powders. Results for iron, Figures 103-107, showed consistently good precision within laboratories, while significant discrepancies were often found between laboratories.

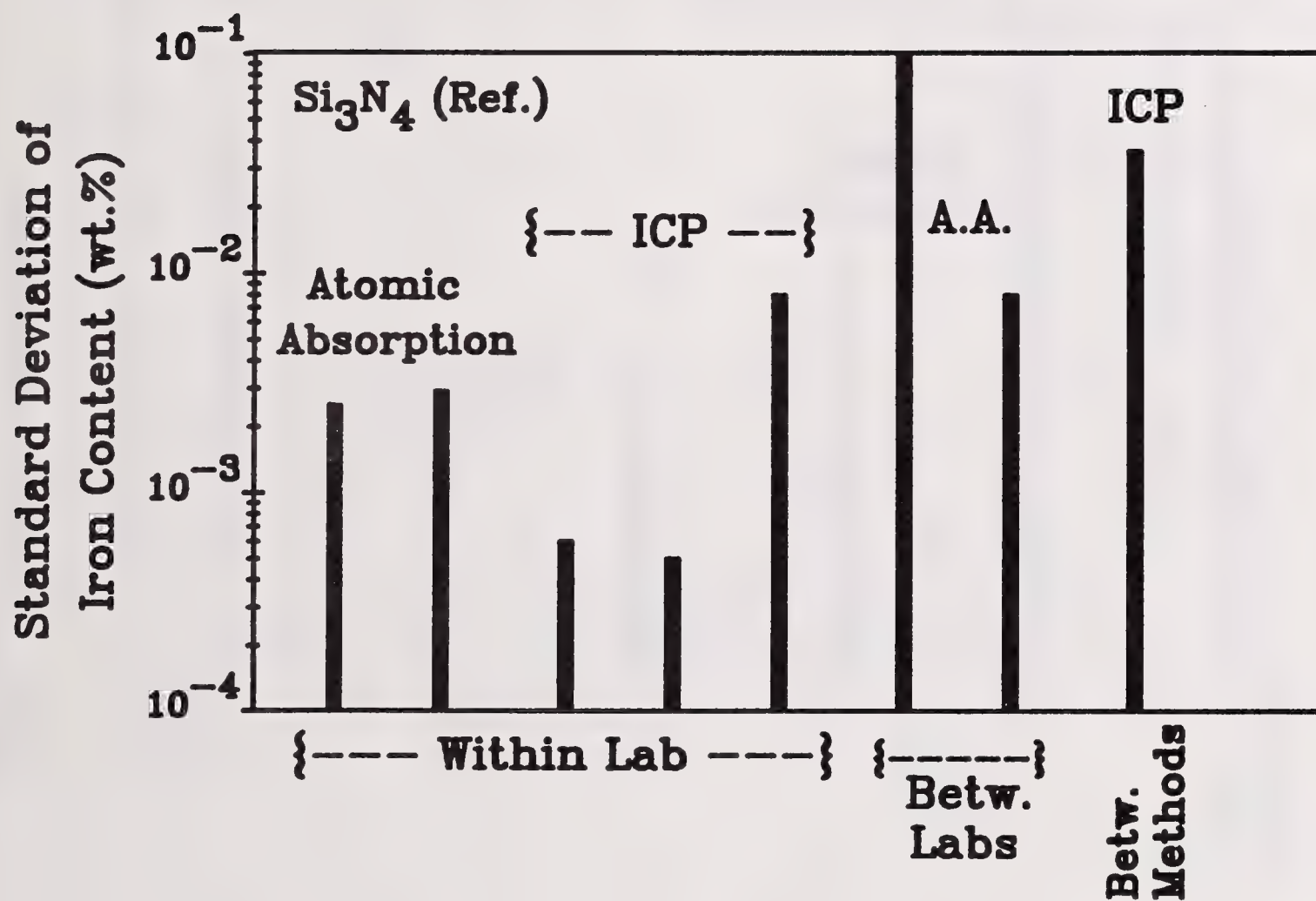


Figure 103.

Within lab, between lab, and between methods standard deviations in the measurement of iron content for the Si_3N_4 (Ref.) powder as measured by various laboratories using a variety of methods.

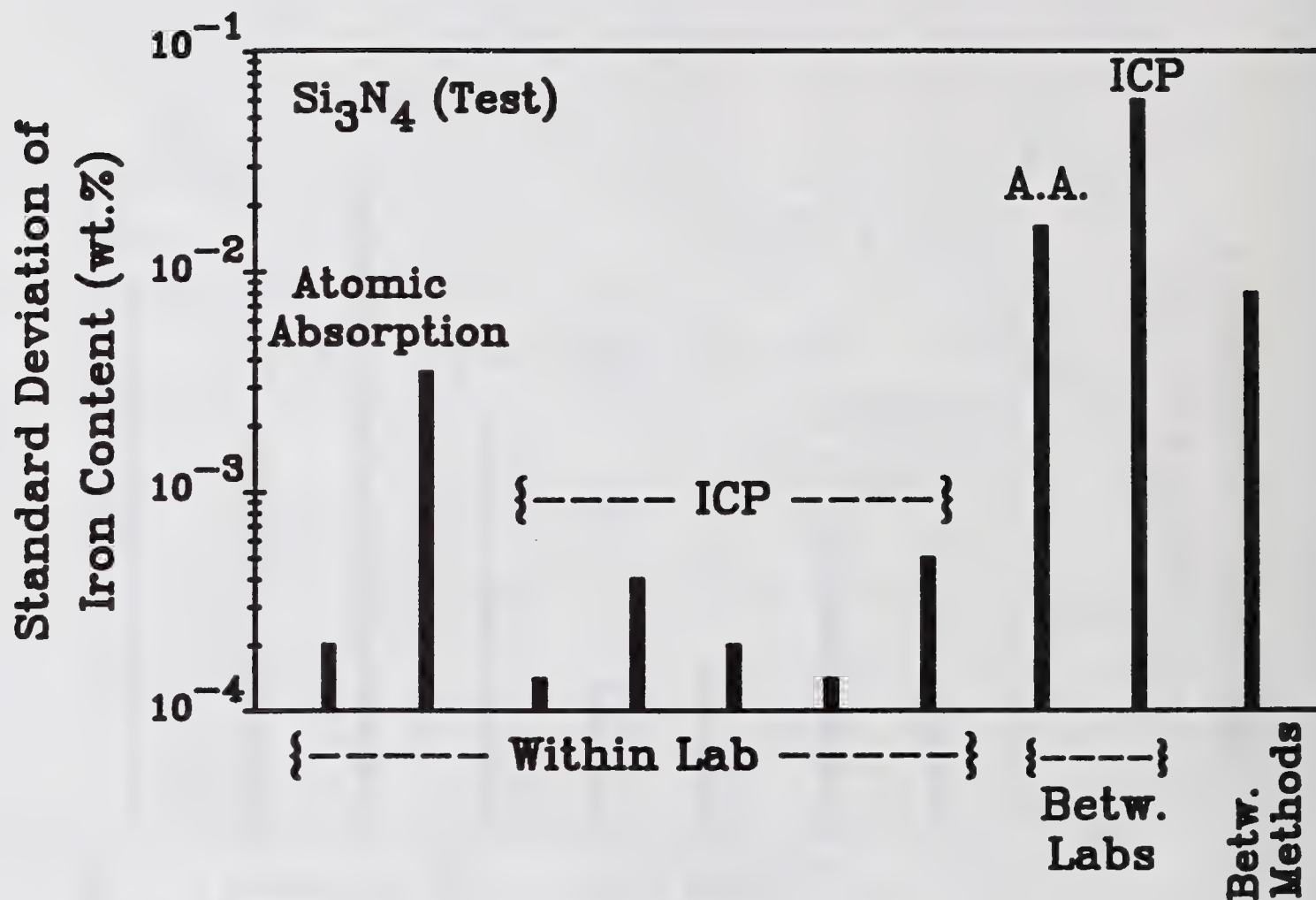


Figure 104.

Within lab, between lab, and between methods standard deviations in the measurement of iron content for the Si_3N_4 (Test) powder as measured by various laboratories using a variety of methods.

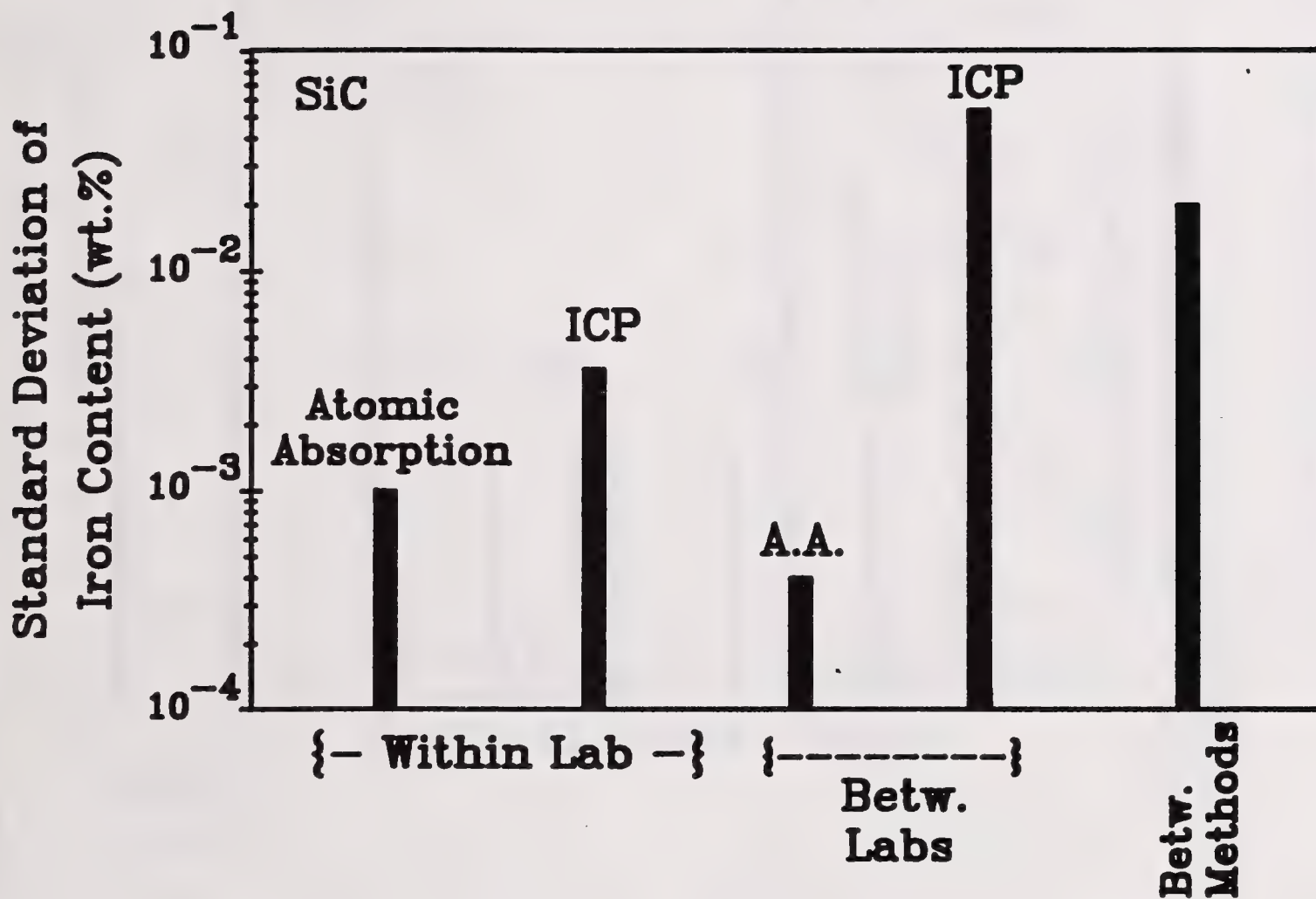


Figure 105.

Within lab, between lab, and between methods standard deviations in the measurement of iron content for the SiC powder as measured by various laboratories using a variety of methods.

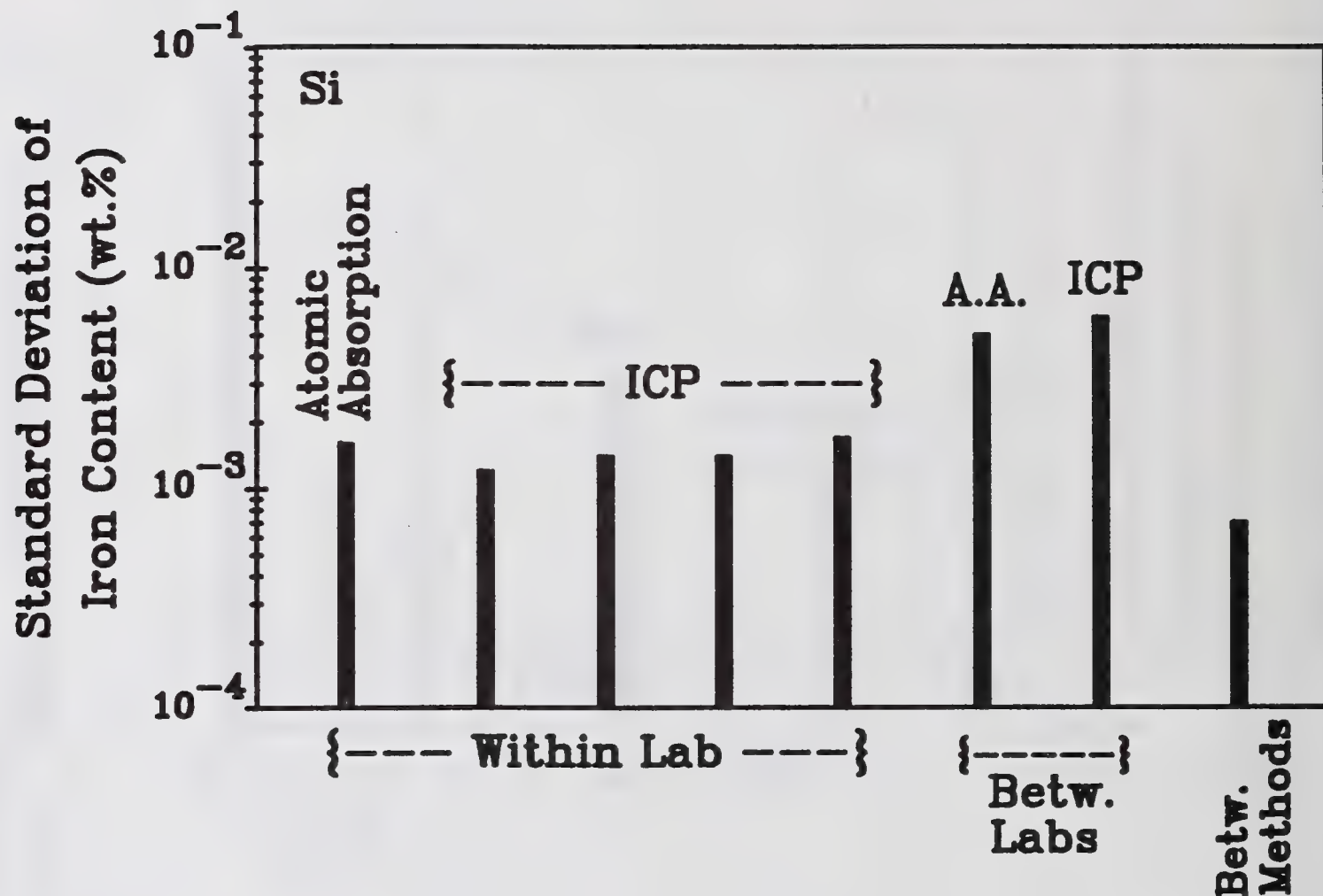


Figure 106.

Within lab, between lab, and between methods standard deviations in the measurement of iron content for the Si powder as measured by various laboratories using a variety of methods.

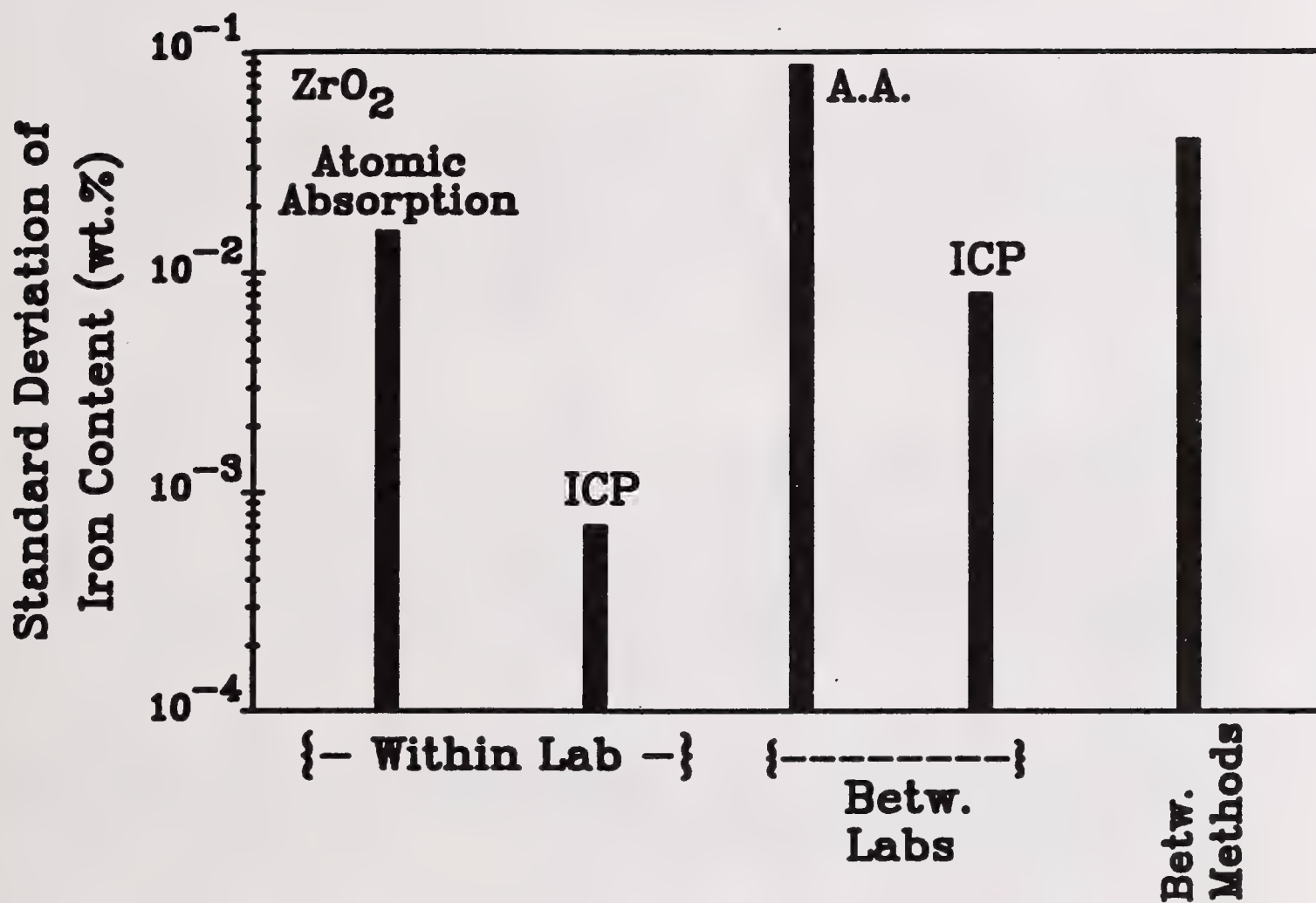


Figure 107.

Within lab, between lab, and between methods standard deviations in the measurement of iron content for the ZrO_2 powder as measured by various laboratories using a variety of methods.

11. PHASE COMPOSITION ANALYSIS

A. L. Dragoo, NIST

11.1. METHODS FOR X-RAY DIFFRACTION

Introduction

X-ray powder diffraction (XRD) is the principal method for the identification of the phase composition of crystalline materials^{1,2}. In recent years, there has been a resurgence of interest in XRD due to the introduction of computer automation diffractometers and processing of x-ray diffraction spectra. Diffractometer automation has provided improved diffractometer performance as well as a reduction in operator time. Computer processing of x-ray data has greatly facilitated phase identification and quantitative analysis of x-ray data.

Scope

XRD is routinely used for qualitative identification of crystalline phases in materials, but quantitative determination as well as evaluation of other material properties, such as crystallite size and residual strain, can be performed readily with modern automated instruments. Electronic computer automation of x-ray diffractometers now provides the capability to perform many aspects of phase identification, quantification, and other property measurements on-line.

XRD can be applied to solid materials in the form of powders, monoliths, fibers etc. However, for greatest accuracy in quantitative analysis, a well-mixed powder sample, prepared in a manner to minimize preferred orientation of grains, is necessary. For quantitative analysis, different approaches for the treatment of data have been devised.

Outline of Method

In an x-ray diffractometer having the Bragg-Brentano geometry, as illustrated in Figure 108, an x-ray beam from a source at F irradiates a specimen S with a divergent, collimated, and sometimes monochromated, beam of x-radiation. The xrays are diffracted by the crystalline planes of the sample and are received by a detector (not shown) which is placed beyond the slits SS in the figure.

The angles through which the xrays are scattered are characteristic of the crystal structure and the intensity of the scattered radiation is characteristic of the atomic composition and the atomic packing of the diffracting planes of atoms.

The detector which is mounted on a goniometer is scanned through a range of angles, measured in units of 2θ , with respect to the source, and simultaneously an angle θ with respect to the sample. The x-radiation used is the characteristic radiation derived from electron bombardment of a metal such as Fe, Cr, Cu, or Mo. The most commonly used radiation is Cu $K\alpha$. This

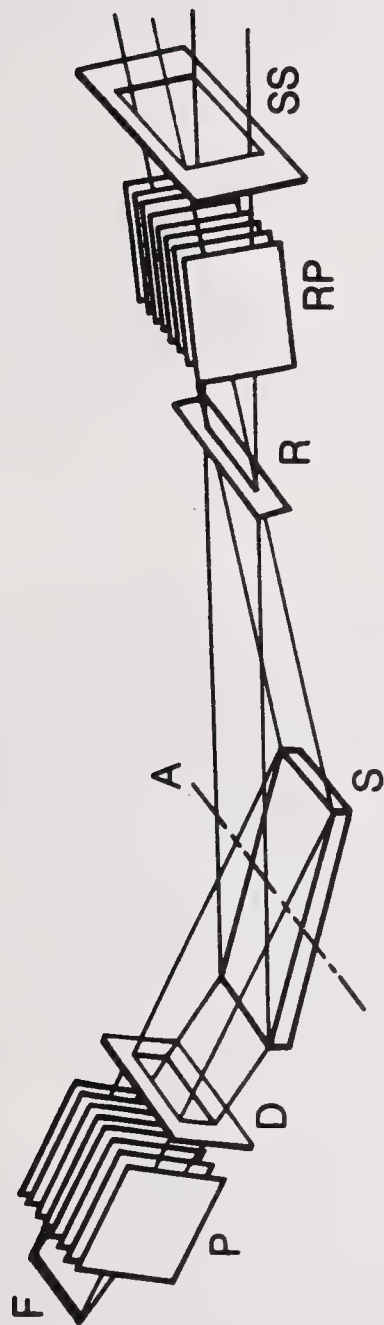


Figure 108. Geometry of the Bragg-Brentano x-ray diffractometer³.

radiation consists of two $K\alpha$ wavelengths due to the α_1 - α_2 doublet and a weaker $K\beta$ wavelength which is removed by use of a Ni filter. The source radiation may be further monochromatized to remove the $K\alpha_2$ wavelength, resulting in high quality incident radiation with nearly a Gaussian line-shape.

Several types of detectors are now in use: Geiger-Müller, scintillation, solid state and position sensitive detectors (PSD). Most detectors are designed to measure the radiation entering through a very small window in 2θ ; consequently, the time required to scan a range of 2θ may require a half an hour or more, depending on the range of 2θ traversed and the scan rate. However, PSD's which were introduced in recent years, use an array of sensitized wires or quartz fibers which are precisely located in the detector to simultaneously record the x-ray beam over a wide range of 2θ . With a PSD, scans of the 2θ spectrum in times less than 15 minutes are now possible.

Qualitative analysis depends on the precise determination of diffraction line positions and, to a lesser extent, on the determination of relative line intensities, for which determination of peak heights usually suffices. Quantitative analysis requires that line intensities also must be accurately measured.

Quantitative Analysis (QXRD)

Quantitative x-ray diffraction (QXRD) analysis of the phase composition of a powder requires careful preparation of samples to minimize the effects of preferred orientation, texture, particle size broadening, and other material effects. Preferred orientation of grains causes erroneously enhanced or reduced intensities with respect to different crystallographic orientations. If coarse particles ($> 10 \mu\text{m}$) are present, peak intensities may be reduced and ragged due to detection of a statistically insufficient number of diffracting grains. Particle size broadening results if grains are finer than $0.1 \mu\text{m}$, leading to a reduction in observed peak height and broadening of diffraction lines. Spray-drying of samples is the optimum method for the preparation of well-mixed samples which do not exhibit preferred orientation, particularly if primary particles are highly acicular. Loading of samples into a sample holder by side-drifting³ may be used successfully if particles have a regular shape. Packed samples should not be used.

The use of integrated line intensities is preferred over the use of peak heights for quantitative analysis. However, integrated intensities often are not used due to the additional experimental difficulty in performing the integration; a task which is frequently encumbered by overlap of significant peaks. Instead, relative concentrations of two or more phases are estimated from heights of one or more isolated peaks of each of the phases present. The peak height method may be further refined by incorporation of a correction factor for one of the phases. Such correction factors may be based on theoretically or empirically obtained coefficients. The fraction, W_i , of the i th phase can be expressed as

$$W_i = K_i I_i / \sum K_j I_j ,$$

where I_i is the intensity (peak height) for phase i and K_i is a coefficient to be determined. Mencik, Short and Peters⁴ developed a method for the analysis of silicon nitride which included the phases α -Si₃N₄, β -Si₃N₄, Si₂ON₂, Si and SiO₂ (α -cristobalite).

Automated processing can now be carried out on the entire electronically collected X-ray diffraction pattern. Rietveld refinement methods are being developed which in addition to material parameters, such as strain, yield the relative concentrations of the crystalline phases.

Quantitative methods used in the present round-robin are summarized in Table 11.1.1.

Standards

Several Standard Reference Materials are available from the National Institute of Standards and Technology for the calibration of x-ray diffraction analyses. These SRM's were reviewed by Dragoo⁵ in 1986. The SRM's are of two types:

- (1) Certified materials for x-ray diffractometer calibration,
- (2) Certified materials for use as line intensity and quantitative analysis standards.

In addition, the JCPDS - International Centre for Diffraction Data^a maintains and disseminates the Powder Diffraction File, which can be accessed electronically, and which provides reference diffraction data (d-spacings, intensities, crystal parameters, etc.) for many phases of interests in ceramics, metallurgy, forensic science, organic and polymer chemistry, and mineralogy.

^aJCPDS - International Centre for Diffraction Data, 1601 Park Lane, Swarthmore, PA 19801.

Table 11.1.1 Procedures for Analysis of X-ray Diffraction Data Obtained in IEA/ANNEX II Powder Characterization Round-Robin.

CODE	PROCEDURE
1	Peak scan, qualitative
2	Peak scan, quantitative, single phase
3	Ratio of peak heights
4	Ratio of integrated intensities
	a. ZrO_2 : Method of Garvie and Nicholson ⁶
5	Modified ratio equation, either peak heights or integrated intensities
	a. $\alpha, \beta\text{-Si}_3\text{N}_4$: Method of Mencik, Short and Peters ⁵
	b. $\alpha, \beta\text{-Si}_3\text{N}_4$: calibration curve
	c. $\alpha, \beta\text{-Si}_3\text{N}_4$: Method of Gazzara and Messier ⁷
	d. ZrO_2 : Modified Garvie-Nicholson method
	(1) Method of Toraya et al. ⁸
	(2) Method of Schmid ⁹
6	Least squares, matrix of intensities
	a. SiC: Peak areas, Lorentzian shape assumed, 8x4 system of coefficients equated to 8 peak areas ¹⁰
7	Whole pattern methods, e.g., Rietveld refinement

Discussion of Results

Data are summarized in the following section.

The results of phase composition determinations by various QXRD methods are summarized in Table 11.1.2. Within the estimated errors of the measurements, little difference is apparent between the various methods used, with the exception of the determination of the β -phase content of the SNT powder using the Rietveld refinement technique. The reason for this difference is not apparent, and further testing of the method, comparisons with other methods, and examination of the powder are required to explain the difference.

Table 11.1.2 Phase Compositions (wt. %) Determined by Various QXRD Methods Uncertainties are Expressed as $\pm$ one Standard Deviation as Determined by the Fitting Procedures of Table 11.1.1

MATERIAL PHASE		METHOD BY CODE (SEE TABLE 11.1.1)					
		1	3	4	5	6	7
SNR	β		6.8 $\pm$ 1.6	4.8 $\pm$.2	5.8 $\pm$.7 5.9 $\pm$.2 ^(a)		5.0 $\pm$.2
	α	Balance					
SNT	β	3.0	4.6 $\pm$.9	2.7 $\pm$.2	3.0 $\pm$.8	3.1 $\pm$.1	9.6 $\pm$.1
SiC	6H	53 ^(b)				80 $\pm$ 5	
	15R	35				13 $\pm$ 4	
	4H	11				5 $\pm$ 1	
	3C	Balance					
YSZ	Tetr.		80 $\pm$ 2	85 $\pm$ 2	81 $\pm$ 5		
	Mono.	Balance					

(a) Calibration curve.

(b) Unspecified method.

11.2. Statistical Data on X-ray Diffraction Analysis

11.2.1. Silicon Nitride Reference Powder (SNR)

Table 11.2.1.1 Beta Phase Content (%) by Ratio of Peak Heights

Material: Silicon Nitride, Reference Powder
 Method: XRD
 Procedure: Ratio of peak heights
 Phase: Beta^a

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
16	6	7.10	8.00	7.47	0.40
21	1	3.00	3.00	3.00	0.00
24	1	7.00	7.00	7.00	0.00
Means	3	3.00	8.00	6.85	1.60

Table 11.2.1.2 Beta Phase Content (%) by Ratio of Intensities

Material: Silicon Nitride, Reference Powder
 Method: XRD
 Procedure: Ratio of Intensities
 Phase: Beta^a

LAB #	# of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
20	2	5.000	5.000	5.00	0.00
22	3	4.566	5.000	4.72	0.24
Means	2	4.566	5.000	4.83	0.23

^a The remainder of the crystalline phases was identified as alpha-Si₃N₄

Table 11.2.1.3 Beta Phase Content (%) by Modified Ratio Equation

Material: Silicon Nitride, Reference Powder
 Method: XRD
 Procedure: Modified ratio equation
 Phase: Beta^a

LAB	\$	\$ of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
2 ^b		1	5.10	5.10	5.10	0.00
4 ^c		1	5.00	5.00	5.00	0.00
5		2	6.20	6.30	6.20	0.07
6		1	5.00	5.00	5.00	0.00
13		11	6.00	7.00	6.18	0.40
15		1	6.00	6.00	6.00	0.00
25		3	4.30	4.70	4.53	0.21
Means		7	4.30	7.00	5.76	0.74

^a The remainder of the crystalline phases was identified as alpha-Si₃N₄, with the exception of Lab No. 2

^b 0.4% Si₂ON₂ also reported

^c Fraction of alpha-Si₃N₄ calculated by independent formula

Table 11.2.1.4 Beta Phase Content (%) by Calibration Curve

Material: Silicon Nitride, Reference Powder
 Method: XRD
 Procedure: Calibration Curve
 Phase: Beta^a

LAB	\$	\$ of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
9		8	5.60	6.10	5.88	0.18
Means		1	5.60	6.10	5.88	0.18

^a The remainder of the crystalline phases were identified as alpha-Si₃N₄ plus Si₂ON₂(trace) and free Si(0.4%)

Table 11.2.1.5 Beta Phase Content (%) by Rietveld Refinement

Material: Silicon Nitride, Reference Powder
 Method: XRD
 Procedure: Whole pattern anal., Rietveld
 Phase: Beta^a

LAB	\$	\$ of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
23		4	4.80	5.21	4.97	0.20
Means		1	4.80	5.21	4.97	0.20

^a The remainder of the crystalline phases was identified as alpha-Si₃N₄

11.2.2. Silicon Nitride Test Powder (SNT)

Table 11.2.2.1 Beta Phase Content (%) by Qualitative Scan of Peak Heights

Material: Silicon Nitride, Test Powder
 Method: XRD
 Procedure: Peak Scan, Qualitative
 Phase: Beta^a

LAB	\$	\$ of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
23		1	3.00	3.00	3.00	0.00
Means		1	3.00	3.00	3.00	0.00

Table 11.2.2.2 Beta Phase Content (%) by Ratio of Peak Heights

Material: Silicon Nitride, Test Powder
 Method: XRD
 Procedure: Ratio of peak heights
 Phase: Beta^a

LAB	\$	\$ of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
14		1	3.10	3.10	3.10	0.00
16		6	4.80	5.50	5.07	0.30
21		1	3.00	3.00	3.00	0.00
24		2	5.00	5.00	5.00	0.00
Means		4	3.00	5.50	4.65	0.87

^a The remainder of the crystalline phases was identified as crystalline alpha-Si₃N₄

Table 11.2.2.3 Beta Phase Content (%) by Ratio of Intensities

Material: Silicon Nitride, Test Powder
 Method: XRD
 Procedure: Ratio of Intensities
 Phase: Beta^a

LAB	\$	\$ of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
20		1	3.00	3.00	3.00	0.00
22		4	2.45	2.83	2.60	0.16
Means		2	2.45	3.00	2.68	0.23

^a The remainder of the crystalline phases was identified as alpha-Si₃N₄

Table 11.2.2.4 Beta Phase Content (%) by Modified Ratio Equation

Material: Silicon Nitride, Test Powder
 Method: XRD
 Procedure: Modified ratio equation
 Phase: Beta^a

LAB	\$	\$ of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
2		1	3.00	3.00	3.00	0.00
4		2	2.00	2.00	2.00	0.00
5		2	3.30	3.60	3.45	0.21
6		1	4.00	4.00	4.00	0.00
15		1	4.00	4.00	4.00	0.00
25		1	2.50	2.50	2.50	0.00
Means		6	2.00	4.00	3.05	0.82

^a The remainder of the crystalline phases was identified as alpha-Si₃N₄

Table 11.2.2.5 Beta Phase Content (%) by Matrix of Intensities

Material: Silicon Nitride, Test Powder
 Method: XRD
 Procedure: Least squares, matrix of intensities
 Phase: Beta^a

LAB	\$	\$ of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
23		2	3.00	3.20	3.10	0.14
Means		1	3.00	3.20	3.10	0.14

^a The remainder of the crystalline phases was identified as alpha-Si₃N₄

Table 11.2.2.6 Beta Phase Content (%) by Rietveld Refinement

Material: Silicon Nitride, Test Powder
 Method: XRD
 Procedure: Whole pattern anal., Rietveld
 Phase: Beta^a

LAB	\$	\$ of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
23		4	9.49	9.80	9.62	0.14
Means		1	9.49	9.80	9.62	0.14

^a The remainder of the crystalline phases was identified as alpha-Si₃N₄

11.2.3. Silicon Carbide (SiC)

Table 11.2.3.1 Phase Content (%) by Matrix of Intensities

Material: Silicon Carbide
 Method: XRD
 Procedure: Least squares, matrix of intensities
 Phase: 6H

LAB	\$	\$ of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
4		2	76.5	77.3	76.9	0.56
6		1	85.0	85.0	85.0	0.00
Means		2	76.5	85.0	79.6	4.69

Phase: 15R

LAB	\$	\$ of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
4		2	13.8	16.4	15.1	1.84
6		1	9.0	9.0	9.0	0.00
Means		2	9.0	16.4	13.1	3.75

Phase: 4H

LAB	\$	\$ of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
4		2	4.0	5.5	4.8	1.06
6		1	6.0	6.0	6.0	0.00
Means		2	4.0	6.0	5.2	1.04

Table 11.2.3.2 Phase Content by Unspecified Method

Material: Silicon Carbide
Method: XRD
Procedure: Not specified
Phase: 6H

LAB	\$	\$ of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
2		1	53.00	53.00	53.00	0.00
Means		1	53.00	53.00	53.00	0.00

Phase: 15R

LAB	\$	\$ of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
2		1	35.0	35.0	35.0	0.00
Means		1	35.0	35.0	35.0	0.00

Phase: 4H

LAB	\$	\$ of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
2		1	11.00	11.00	11.00	0.00
Means		1	11.00	11.00	11.00	0.00

11.2.4. Yttria-Stabilized Zirconia (YSZ)

Table 11.2.4.1 Phase Content (%) by Ratio of Peak Heights

Material: Zirconia
Method: XRD
Procedure: Ratio of peak heights
Phase: Tetragonal

LAB	\$	\$ of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
2		1	82.0	82.0	82.0	0.0
14		1	80.0	80.0	80.0	0.0
24		3	78.0	81.0	79.3	1.5
Means		3	78.0	82.0	80.0	1.6

Phase: Monoclinic

LAB	\$	\$ of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
2		1	18.0	18.0	18.0	0.0
14		1	20.0	20.0	20.0	0.0
24		3	19.0	22.0	20.7	1.5
Means		3	18.0	22.0	20.0	1.6

Table 11.2.4.2 Phase Content by Ratio of Intensities

Material: Zirconia
Method: XRD
Procedure: Ratio of intensities
Phase: Tetragonal

LAB	\$	\$ of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
9		3	84.40	84.90	84.57	0.29
22		1	87.92	87.92	87.92	0.00
Means		2	84.40	87.92	85.40	1.69

Phase: Monoclinic

LAB	\$	\$ of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
9		3	15.10	15.60	15.43	0.29
22		1	12.08	12.08	12.08	0.00
Means		2	12.08	15.60	14.60	1.69

Table 11.2.4.3 Phase Content by Modified Ratio Equation

Material: Zirconia
 Method: XRD
 Procedure: Modified ratio equation
 Phase: Tetragonal

LAB	\$	\$ of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
6		1	90.6	90.6	90.6	0.00
15		1	83.0	83.0	83.0	0.00
18		6	75.9	80.0	78.6	1.76
Means		3	75.9	90.6	80.7	4.54

Phase: Monoclinic

LAB	\$	\$ of Values	Minimum Value	Maximum Value	Average Value	Standard Deviation
6		1	9.4	9.4	9.4	0.00
15		1	17.0	17.0	17.0	0.00
18		6	20.0	24.1	21.4	1.76
Means		3	9.4	24.1	19.3	4.54

11.3. References

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12. THERMAL ANALYSIS

A. L. Dragoo and S. G. Malghan, NIST

Introduction

Thermal analysis includes a variety of techniques to measure thermochemical and thermomechanical properties of materials, such as differential thermal analysis (DTA), calorimetry, dilatometry and thermal gravimetric analysis (TGA). For this study, a very few laboratories reported results obtained using TGA to measure the amount of volatile components in the powders.

12.1. Results

The amounts of volatile components of the powders are illustrated here with the results reported by Lab 4.

Table 12.1 TGA Mass Loss (%) as Determined by Lab 4. Heating rate 5 °C/min; ambient air; sample on Pt pan

POWDER	SAMPLE WT., mg	SAMPLE CHARACTERISTICS	T, °C	MASS LOSS
SNR	63.7	Granules	150	0.28
			400	No change
	42.7	Granules	400	0.23
			400	No change
SNT	102.8	Large granule	150	0.14
			250	0.17
	43.7	Large granule	150	0.19
SiC	110.8	Loose pellet	150	0.14
			350	0.27
Si	65.8	Not specified	150	0.07
YSZ	74.3		400	0.54

APPENDIX I - Physical Properties

Table I.1 lists the methods, code numbers, and units used in the measurement of density and specific surface area of the powders. The code numbers identify the methods tabulated in Table I.2 (density) and in Table I.3 (specific surface area).

Table I.1. Methods and Codes for Physical Property Measurements

<u>Method</u>	<u>Code</u>	<u>Units</u>
Single Point Surface Area	1	m ² /g
Multi Point Surface Area	2	m ² /g
Tap Density	3	g/cm ³
He gas pycnometer	4	g/cm ³
Liquid pycnometer, hydrocarbon	6	g/cm ³
Density, Hg Intrusion	13	g/cm ³

Table I.2 Density

I.2.1.

Silicon Nitride Reference Powder

Lab #	IEA Vial #	Method Code	Tap Density	Bulk Density	Liquid Density
5	9001	3	0.74	---	---
5	9004	3	0.78	---	---
6	9019	3	0.87	---	---
15	9013	3	0.77	---	---
15	9013	3	0.77	---	---
17	225	3	1.46	---	---
17	1898	3	1.47	---	---
17	1898	3	1.48	---	---
18	9024	3	0.74	---	---
21	9007	3	0.71	---	---
25	9010	3	0.79	---	---
17	980102	4	---	3.130	---
17	980102	4	---	3.150	---
17	980102	4	---	3.130	---
17	980102	4	---	3.130	---
17	980102	4	---	3.150	---
17	2410338	4	---	3.140	---
17	2410338	4	---	3.120	---
17	2410338	4	---	3.130	---
17	2410338	4	---	3.120	---
17	2410338	4	---	3.130	---
17	3770991	4	---	3.180	---
17	3770991	4	---	3.180	---
17	3770991	4	---	3.160	---
17	3770991	4	---	3.180	---
17	3770991	4	---	3.160	---
17	7757988	4	---	3.130	---
17	7757988	4	---	3.130	---
17	7757988	4	---	3.130	---
17	7757988	4	---	3.120	---
17	7757988	4	---	3.120	---
17	9461894	4	---	3.124	---
17	9461894	4	---	3.124	---
17	9461894	4	---	3.124	---
17	9461894	4	---	3.137	---
17	9461894	4	---	3.137	---
23	16	4	---	3.132	---
23	16	4	---	3.152	---
23	16	4	---	3.136	---
23	377	4	---	3.131	---
23	377	4	---	3.146	---
23	377	4	---	3.157	---
23	16010813	4	---	3.141	---

Table I.2 Density (Continue)

I.2.1.

Silicon Nitride Reference Powder

Lab #	IEA Vial #	Method Code	Tap Density	Bulk Density	Liquid Density
21	9007	6	---	---	0.56
25	9010	6	---	---	0.48

I.2.2.

Silicon Nitride Test Powder

Lab #	IEA Vial #	Method Code	Tap Density	Bulk Density	Liquid Density
6	18	3	0.76	---	---
14	LSM06	3	0.67	---	---
14	LSM07	3	0.65	---	---
15	LSM09	3	0.66	---	---
18	26	3	0.71	---	---
21	LSM05	3	0.66	---	---
25	LSM03	3	0.71	---	---
17	1350810	4	---	3.150	---
17	1350810	4	---	3.130	---
17	1350810	4	---	3.160	---
17	1350810	4	---	3.180	---
17	1350810	4	---	3.180	---
17	1350810	4	---	3.180	---
17	1350810	4	---	3.180	---
17	1471273	4	---	3.160	---
17	1471273	4	---	3.140	---
17	1471273	4	---	3.140	---
17	1471273	4	---	3.160	---
17	1471273	4	---	3.160	---
17	1800522	4	---	3.160	---
17	1800522	4	---	3.160	---
17	1800522	4	---	3.160	---
17	1800522	4	---	3.160	---
17	1800522	4	---	3.160	---
17	2831011	4	---	3.160	---
17	2831011	4	---	3.190	---
17	2831011	4	---	3.180	---
17	2831011	4	---	3.160	---
17	2831011	4	---	3.130	---
17	3410361	4	---	3.160	---
17	3410361	4	---	3.190	---
17	3410361	4	---	3.170	---
17	3410361	4	---	3.200	---
17	3410361	4	---	3.170	---
17	3410361	4	---	3.170	---
17	8400644	4	---	3.160	---
17	8400644	4	---	3.150	---
17	8400644	4	---	3.150	---
17	8400644	4	---	3.160	---
17	8400644	4	---	3.150	---
17	11571041	4	---	3.160	---

Silicon Nitride Test Powder

Lab #	IEA Vial #	Method Code	Tap Density	Bulk Density	Liquid Density
17	11571041	4	---	3.160	---
17	11571041	4	---	3.160	---
17	11571041	4	---	3.150	---
17	11571041	4	---	3.180	---
17	12571240	4	---	3.150	---
17	12571240	4	---	3.160	---
17	12571240	4	---	3.150	---
17	12571240	4	---	3.160	---
17	12571240	4	---	3.160	---
23	1913	4	---	3.144	---
23	U0904	4	---	3.170	---
23	U0904	4	---	3.164	---
23	U0904	4	---	3.144	---
23	U1913	4	---	3.138	---
23	U1913	4	---	3.152	---
23	U1913	4	---	3.136	---
25	LSM03	6	---	---	0.43

I.2.3.

Silicon Carbide Powder

Lab #	IEA Vial #	Method Code	Tap Density	Bulk Density	Liquid Density
6	1003	3	0.83	---	---
10	1018	3	0.71	---	---
10	1018	3	1.72	---	---
10	1018	3	1.71	---	---
10	1018	3	1.71	---	---
10	1018	3	1.72	---	---
10	1018	3	1.71	---	---
10	1018	3	1.76	---	---
10	1018	3	1.76	---	---
10	1018	3	1.77	---	---
10	1018	3	1.76	---	---
10	1018	3	1.76	---	---
10	1018	3	1.80	---	---
10	1018	3	1.79	---	---
10	1018	3	1.79	---	---
10	1018	3	1.77	---	---
10	1018	3	1.78	---	---
15	1012	3	0.77	---	---
18	1009	3	0.68	---	---
21	1011	3	0.76	---	---
21	1011	3	0.75	---	---
21	1011	3	0.75	---	---
25	1016	3	0.78	---	---
17	1160117	4	---	3.170	---
17	1160117	4	---	3.180	---
17	1160117	4	---	3.150	---
17	1160117	4	---	3.180	---
17	1160117	4	---	3.150	---
17	1200119	4	---	3.130	---
17	1200119	4	---	3.130	---
17	1200119	4	---	3.120	---
17	1200119	4	---	3.120	---
17	1200119	4	---	3.110	---
17	1210118	4	---	3.150	---
17	1210118	4	---	3.150	---
17	1210118	4	---	3.160	---
17	1210118	4	---	3.160	---
17	1210118	4	---	3.150	---
17	1220123	4	---	3.110	---
17	1220123	4	---	3.080	---

Silicon Carbide Powder

Lab #	IEA Vial #	Method Code	Tap Density	Bulk Density	Liquid Density
17	1220123	4	---	3.130	---
17	1220123	4	---	3.130	---
17	1220123	4	---	3.130	---
17	1330134	4	---	3.140	---
17	1330134	4	---	3.124	---
17	1330134	4	---	3.124	---
17	1330134	4	---	3.171	---
17	1330134	4	---	3.155	---
17	1353637	4	---	3.160	---
17	1353637	4	---	3.160	---
17	1353637	4	---	3.160	---
17	1353637	4	---	3.170	---
17	1353637	4	---	3.150	---
17	1390138	4	---	3.160	---
17	1390138	4	---	3.120	---
17	1390138	4	---	3.150	---
17	1390138	4	---	3.150	---
17	1390138	4	---	3.190	---
17	1390138	4	---	3.180	---
23	1005	4	---	3.160	---
23	1005	4	---	3.160	---
23	1005	4	---	3.160	---
23	1005	4	---	3.160	---
23	1005	4	---	3.160	---
23	1005	4	---	3.140	---
23	1005	4	---	3.140	---
23	1005	4	---	3.140	---
23	1005	4	---	3.140	---
23	1005	4	---	3.140	---
23	1005	4	---	3.140	---
23	1005	4	---	3.140	---
23	1005	4	---	3.158	---
23	1005	4	---	3.124	---
23	1005	4	---	3.137	---
23	1703	4	---	3.136	---
23	1703	4	---	3.151	---
23	1703	4	---	3.157	---
25	1016	6	---	---	0.40

I.2.4.

Silicon Powder

Lab #	IEA Vial #	Method Code	Tap Density	Bulk Density	Liquid Density
6	1003	3	1.08	---	---
15	1016	3	1.03	---	---
17	43	3	1.47	---	---
17	43	3	1.48	---	---
17	44	3	1.46	---	---
18	1015	3	0.78	---	---
17	86087	4	---	2.310	---
17	86087	4	---	2.310	---
17	86087	4	---	2.320	---
17	86087	4	---	2.300	---
17	86087	4	---	2.310	---
17	88089	4	---	2.320	---
17	88089	4	---	2.320	---
17	88089	4	---	2.320	---
17	88089	4	---	2.320	---
17	88089	4	---	2.320	---
17	91092	4	---	2.330	---
17	91092	4	---	2.330	---
17	91092	4	---	2.340	---
17	91092	4	---	2.330	---
17	91092	4	---	2.330	---
17	1170128	4	---	2.340	---
17	1170128	4	---	2.340	---
17	1170128	4	---	2.330	---
17	1170128	4	---	2.350	---
17	1170128	4	---	2.340	---
17	1180119	4	---	2.308	---
17	1180119	4	---	2.324	---
17	1180119	4	---	2.300	---
17	1180119	4	---	2.308	---
17	1180119	4	---	2.324	---
17	1270116	4	---	2.350	---
17	1270116	4	---	2.350	---
17	1270116	4	---	2.340	---
17	1270116	4	---	2.350	---
17	1270116	4	---	2.340	---
17	1300129	4	---	2.340	---
17	1300129	4	---	2.330	---
17	1300129	4	---	2.340	---
17	1300129	4	---	2.330	---
17	1300129	4	---	2.330	---

Silicon Powder

Lab #	IEA Vial #	Method Code	Tap Density	Bulk Density	Liquid Density
17	1310132	4	---	2.310	---
17	1310132	4	---	2.310	---
17	1310132	4	---	2.320	---
17	1310132	4	---	2.320	---
17	1310132	4	---	2.310	---
23	1005	4	---	2.344	---
23	1005	4	---	2.336	---
23	1005	4	---	2.335	---
23	2203	4	---	2.340	---
23	2203	4	---	2.330	---
23	2203	4	---	2.330	---
23	2203	4	---	2.330	---
23	2203	4	---	2.330	---
23	2204	4	---	2.330	---
23	2204	4	---	2.330	---
23	2204	4	---	2.330	---
23	2204	4	---	2.330	---
23	2204	4	---	2.330	---
23	2204	4	---	2.333	---
23	2204	4	---	2.328	---
23	2204	4	---	2.342	---

I.2.5.

Yttria-Zirconia Powder

Lab #	IEA Vial #	Method Code	Tap Density	Bulk Density	Liquid Density
6	OE013	3	1.52	---	---
14	OC01	3	1.50	---	---
14	OC01	3	1.50	---	---
15	OE014	3	1.49	---	---
18	259	3	1.48	---	---
18	534	3	1.46	---	---
18	OR012	3	1.47	---	---
17	59	4	---	5.666	---
17	59	4	---	5.631	---
17	76	4	---	5.675	---
17	76	4	---	5.700	---
17	76	4	---	5.685	---
17	121	4	---	5.810	---
17	121	4	---	5.734	---
17	380	4	---	5.750	---
17	380	4	---	5.606	---
17	510	4	---	5.631	---
17	510	4	---	5.717	---
17	592	4	---	5.636	---
17	592	4	---	5.650	---
17	592	4	---	5.727	---
18	39	4	---	6.001	---
18	OR012	4	---	5.910	---
18	OR012	4	---	5.978	---
18	OR012	4	---	5.903	---
14	OC01	6	---	---	1.24
14	OC01	6	---	---	1.22
22	269	13	---	6.467	---
22	269	13	---	7.139	---
22	269	13	---	7.211	---
22	279	13	---	6.020	---
22	279	13	---	6.068	---
22	279	13	---	7.566	---

Table I.3 SPECIFIC SURFACE AREA

I.3.1. Silicon Nitride Reference Powder

Lab #	IEA Vial #	Method Code	Single- Point	Multi- Point
2	119	1	13.20	---
3	542	1	11.80	---
3	556	1	11.60	---
3	557	1	11.60	---
4	438	1	13.80	---
4	438	1	13.90	---
4	455	1	13.70	---
4	455	1	13.80	---
4	455	1	13.90	---
4	977	1	13.30	---
4	977	1	13.40	---
4	999	1	13.17	---
4	999	1	13.21	---
4	999	1	13.14	---
4	999	1	13.14	---
4	1003	1	13.10	---
4	1003	1	13.10	---
4	1003	1	13.11	---
4	1003	1	13.14	---
5	1631	1	12.40	---
8	984	1	14.04	---
8	984	1	14.02	---
8	984	1	14.00	---
8	984	1	14.03	---
11	9003	1	13.70	---
14	100	1	14.30	---
14	100	1	14.80	---
14	305	1	13.60	---
14	307	1	14.20	---
14	770	1	14.00	---
15	9013	1	12.90	---
15	9013	1	13.10	---
16	638	1	13.70	---
16	638	1	14.00	---
16	686	1	13.60	---
16	686	1	13.80	---
16	688	1	13.60	---
16	688	1	13.80	---
16	688	1	13.80	---
17	48	1	12.94	---

Silicon Nitride Reference Powder

Lab #	IEA Vial #	Method Code	Single- Point	Multi- Point
17	48	1	12.98	---
17	48	1	13.13	---
17	48	1	12.94	---
17	48	1	12.94	---
17	225	1	13.18	---
17	225	1	13.09	---
17	225	1	13.09	---
17	225	1	13.36	---
17	582	1	12.88	---
17	582	1	12.88	---
17	582	1	12.92	---
17	582	1	12.88	---
17	582	1	12.79	---
17	582	1	12.79	---
17	582	1	12.79	---
17	582	1	12.74	---
17	719	1	13.07	---
17	719	1	12.87	---
17	719	1	12.99	---
17	719	1	12.91	---
17	761	1	12.85	---
17	761	1	12.80	---
17	761	1	12.74	---
17	761	1	12.85	---
17	987	1	12.55	---
17	987	1	12.44	---
17	987	1	12.55	---
17	987	1	12.44	---
17	1104	1	13.59	---
17	1104	1	13.71	---
17	1104	1	13.59	---
17	1104	1	13.67	---
17	1111	1	12.78	---
17	1111	1	12.89	---
17	1111	1	12.67	---
17	1111	1	12.82	---
17	1117	1	12.82	---
17	1117	1	12.82	---
17	1117	1	12.72	---
17	1117	1	12.82	---
17	1164	1	12.72	---
17	1164	1	12.72	---

Silicon Nitride Reference Powder

Lab #	IEA Vial #	Method Code	Single- Point	Multi- Point
17	1164	1	12.66	---
17	1164	1	12.61	---
17	1222	1	13.32	---
17	1222	1	13.28	---
17	1222	1	13.28	---
17	1222	1	13.37	---
17	1222	1	13.32	---
17	1381	1	12.73	---
17	1381	1	12.85	---
17	1381	1	12.65	---
17	1381	1	12.81	---
17	1381	1	12.93	---
17	1381	1	12.77	---
17	1381	1	12.82	---
17	1381	1	12.82	---
17	1381	1	12.98	---
17	1381	1	12.93	---
17	1381	1	13.04	---
17	1381	1	13.10	---
17	1381	1	12.93	---
17	1516	1	13.00	---
17	1516	1	13.13	---
17	1516	1	13.05	---
17	1516	1	13.18	---
17	1516	1	13.18	---
17	1538	1	12.70	---
17	1538	1	12.66	---
17	1538	1	12.78	---
17	1538	1	12.78	---
17	1592	1	13.58	---
17	1592	1	13.47	---
17	1592	1	13.58	---
17	1592	1	13.52	---
17	1898	1	13.01	---
17	1898	1	13.08	---
17	1898	1	13.01	---
17	1898	1	13.04	---
18	1032	1	12.45	---
18	1032	1	12.87	---
18	1032	1	12.70	---
18	1032	1	13.04	---
18	1032	1	12.97	---

Silicon Nitride Reference Powder

Lab #	IEA Vial #	Method Code	Single- Point	Multi- Point
18	1032	1	12.82	---
18	1032	1	13.02	---
18	1032	1	12.93	---
18	1032	1	13.03	---
24	244	1	12.00	---
24	648	1	12.60	---
24	9005	1	12.08	---
24	9005	1	12.25	---
24	9005	1	12.19	---
25	1213	1	13.10	---
25	1231	1	13.10	---
25	1869	1	13.20	---
1	599	2	---	14.51
1	1255	2	---	14.20
5	1631	2	---	13.20
6	1857	2	---	12.93
7	302	2	---	13.47
7	1519	2	---	13.56
9	159	2	---	15.35
9	697	2	---	14.45
9	703	2	---	13.72
9	1173	2	---	14.99
9	1752	2	---	15.13
13	56	2	---	13.54
13	253	2	---	14.61
13	375	2	---	12.80
13	400	2	---	12.90
13	624	2	---	12.80
13	1016	2	---	12.61
13	1113	2	---	11.29
13	1281	2	---	13.04
13	1315	2	---	12.89
13	1880	2	---	13.10
14	305	2	---	12.30
14	305	2	---	12.50
14	307	2	---	13.00
14	307	2	---	13.10
20	198	2	---	13.09
20	637	2	---	13.23
20	652	2	---	13.48
20	1241	2	---	12.92

Silicon Nitride Reference Powder

Lab #	IEA Vial #	Method Code	Single- Point	Multi- Point
20	1394	2	---	13.43
20	1394	2	---	13.03
20	1695	2	---	13.34
21	24	2	---	13.61
21	24	2	---	13.59
22	130	2	---	12.92
22	130	2	---	13.10
22	130	2	---	15.53
22	130	2	---	15.40
22	1449	2	---	12.91
22	1449	2	---	12.99
22	1449	2	---	12.24
22	1449	2	---	13.59
22	1449	2	---	13.94
22	1578	2	---	13.00
22	1578	2	---	13.11
22	1578	2	---	12.07
22	1578	2	---	14.20
22	1578	2	---	14.15
23	1009	2	---	13.20
23	1009	2	---	13.40
23	1009	2	---	13.49
23	1022	2	---	13.25
23	1022	2	---	13.21
23	1022	2	---	13.27
23	1410	2	---	13.26
23	1410	2	---	13.44
23	1410	2	---	13.62
23	1471	2	---	13.33
23	1471	2	---	13.80
23	1471	2	---	13.65
23	1471	2	---	12.94
23	1471	2	---	12.75
23	1471	2	---	13.83
23	1682	2	---	13.89
23	1682	2	---	13.63
23	1682	2	---	13.63
23	1812	2	---	13.41
23	1812	2	---	13.39
23	1812	2	---	13.21

I.3.2.

Silicon Nitride Test Powder

Lab #	IEA Vial #	Method Code	Single- Point	Multi- Point
2	446	1	9.10	---
2	604	1	9.10	---
3	213	1	8.49	---
3	1302	1	8.44	---
4	31	1	9.83	---
4	31	1	9.81	---
4	31	1	9.83	---
4	31	1	9.81	---
4	730	1	9.83	---
4	730	1	9.82	---
4	730	1	9.76	---
4	730	1	9.72	---
4	1427	1	9.79	---
4	1427	1	9.79	---
4	1427	1	9.79	---
4	1427	1	9.81	---
5	818	1	9.20	---
5	818	1	9.20	---
5	821	1	9.10	---
5	821	1	9.10	---
8	223	1	10.33	---
8	223	1	10.28	---
8	223	1	10.34	---
8	353	1	10.21	---
8	353	1	10.24	---
8	353	1	10.24	---
8	353	1	10.21	---
8	1417	1	10.43	---
8	1417	1	10.30	---
8	1417	1	10.30	---
14	1182	1	11.00	---
14	1182	1	11.00	---
14	1182	1	9.00	---
15	LSM09	1	8.70	---
16	1036	1	9.25	---
16	1036	1	9.29	---
16	1036	1	9.25	---
16	1199	1	8.92	---
16	1199	1	8.88	---
16	1199	1	9.03	---
17	312	1	9.22	---
17	312	1	9.30	---

Silicon Nitride Test Powder

Lab #	IEA Vial #	Method Code	Single- Point	Multi- Point
17	312	1	9.13	---
17	312	1	9.26	---
17	312	1	9.26	---
17	312	1	8.67	---
17	312	1	8.90	---
17	312	1	8.85	---
17	312	1	8.67	---
17	384	1	9.44	---
17	384	1	9.36	---
17	384	1	9.48	---
17	384	1	9.44	---
17	680	1	8.62	---
17	680	1	8.73	---
17	680	1	8.68	---
17	680	1	8.68	---
17	938	1	8.99	---
17	938	1	8.86	---
17	938	1	8.76	---
17	938	1	8.96	---
17	938	1	9.03	---
17	938	1	8.99	---
17	938	1	8.91	---
17	938	1	8.99	---
17	1315	1	9.13	---
17	1315	1	9.16	---
17	1315	1	9.16	---
17	1315	1	9.10	---
17	1419	1	9.04	---
17	1419	1	9.04	---
17	1419	1	9.13	---
17	1419	1	9.13	---
17	1419	1	8.84	---
17	1419	1	8.92	---
17	1419	1	8.96	---
17	1419	1	8.84	---
17	1419	1	8.96	---
18	597	1	9.50	---
18	597	1	9.83	---
18	597	1	9.65	---
18	597	1	9.47	---
18	597	1	9.31	---
18	597	1	9.21	---

Silicon Nitride Test Powder

Lab #	IEA Vial #	Method Code	Single-Point	Multi-Point
18	597	1	9.46	---
18	597	1	9.50	---
18	597	1	9.53	---
20	193	1	10.15	---
20	193	1	10.00	---
25	432	1	9.55	---
25	758	1	9.53	---
6	889	2	---	9.31
7	564	2	---	9.89
7	564	2	---	10.63
7	564	2	---	10.49
7	976	2	---	9.83
7	976	2	---	10.62
7	976	2	---	10.53
9	23	2	---	9.77
9	23	2	---	10.04
9	23	2	---	9.86
9	23	2	---	9.17
9	200	2	---	9.68
9	200	2	---	10.09
9	200	2	---	9.90
9	200	2	---	9.33
9	1044	2	---	9.64
9	1044	2	---	9.99
9	1044	2	---	9.81
9	1044	2	---	8.97
9	1152	2	---	10.02
9	1152	2	---	9.81
9	1152	2	---	9.84
9	1152	2	---	9.39
9	1156	2	---	10.07
9	1156	2	---	10.09
9	1156	2	---	9.87
9	1156	2	---	9.36
14	323	2	---	9.50
14	323	2	---	9.50
14	1182	2	---	8.80
14	1182	2	---	9.20
20	252	2	---	10.00
21	LSM05	2	---	9.84
21	LSM05	2	---	9.77

Silicon Nitride Test Powder

Lab #	IEA Vial #	Method Code	Single- Point	Multi- Point
21	LSM05	2	---	10.03
22	915	2	---	10.01
22	915	2	---	10.21
22	915	2	---	10.00
22	915	2	---	9.95
22	1168	2	---	9.87
22	1168	2	---	9.81
22	1168	2	---	10.09
22	1168	2	---	10.04
23	259	2	---	9.52
23	259	2	---	9.54
23	259	2	---	9.67
23	371	2	---	9.75
23	371	2	---	9.81
23	371	2	---	9.75
23	608	2	---	9.71
23	608	2	---	9.62
23	608	2	---	9.90
23	632	2	---	9.83
23	632	2	---	9.64
23	632	2	---	9.71
23	654	2	---	9.53
23	654	2	---	9.46
23	654	2	---	9.52
23	1012	2	---	9.47
23	1012	2	---	9.69
23	1012	2	---	9.69

I.3.3.

Silicon Carbide Powder

Lab #	IEA Vial #	Method Code	Single-Point	Multi-Point
1	241	1	12.40	---
1	241	1	12.53	---
1	243	1	12.36	---
1	243	1	12.50	---
2	22	1	12.80	---
3	4	1	12.40	---
3	4	1	12.30	---
3	4	1	12.60	---
3	7	1	12.70	---
3	7	1	12.80	---
3	7	1	12.90	---
3	9	1	12.90	---
3	9	1	12.90	---
3	9	1	12.90	---
3	11	1	12.80	---
3	11	1	12.70	---
3	11	1	12.70	---
4	73	1	13.75	---
4	73	1	13.77	---
4	73	1	13.79	---
4	73	1	13.74	---
4	76	1	13.70	---
4	76	1	13.69	---
4	76	1	13.63	---
4	76	1	13.66	---
4	77	1	13.68	---
4	77	1	13.68	---
4	77	1	13.79	---
4	77	1	13.78	---
8	268	1	16.69	---
8	268	1	16.49	---
8	268	1	16.79	---
8	271	1	15.49	---
8	271	1	15.46	---
8	271	1	15.41	---
15	1012	1	11.92	---
16	78	1	12.60	---
16	82	1	12.60	---
16	LSM1013	1	12.90	---
17	148	1	13.00	---
17	148	1	12.96	---
17	148	1	13.00	---

Silicon Carbide Powder

Lab #	IEA Vial #	Method Code	Single-Point	Multi-Point
17	148	1	13.00	---
17	149	1	13.00	---
17	149	1	13.04	---
17	149	1	13.00	---
17	149	1	13.00	---
17	150	1	12.96	---
17	150	1	13.10	---
17	150	1	13.19	---
17	150	1	13.19	---
17	150	1	12.96	---
17	152	1	13.12	---
17	152	1	13.12	---
17	152	1	13.20	---
17	152	1	13.20	---
17	153	1	13.06	---
17	153	1	13.09	---
17	153	1	13.06	---
17	153	1	13.09	---
17	154	1	12.99	---
17	154	1	12.94	---
17	154	1	12.94	---
17	154	1	12.99	---
17	154	1	12.99	---
17	154	1	13.07	---
17	154	1	13.07	---
17	154	1	13.07	---
17	154	1	13.09	---
17	154	1	12.99	---
17	154	1	13.04	---
17	154	1	13.04	---
18	94	1	12.36	---
18	94	1	14.05	---
18	94	1	13.15	---
18	94	1	13.38	---
18	94	1	14.00	---
18	94	1	13.27	---
18	94	1	13.27	---
18	94	1	13.82	---
18	94	1	13.10	---
20	90	1	14.31	---
25	1016	1	13.70	---

Silicon Carbide Powder

Lab #	IEA Vial #	Method Code	Single- Point	Multi- Point
1	241	2	---	13.66
1	241	2	---	13.85
1	243	2	---	13.58
1	243	2	---	13.77
6	100	2	---	11.67
7	248	2	---	16.38
7	248	2	---	15.92
7	248	2	---	16.30
9	255	2	---	14.71
9	255	2	---	14.58
9	255	2	---	14.68
9	256	2	---	14.78
9	256	2	---	14.66
9	256	2	---	14.52
9	261	2	---	14.50
9	261	2	---	14.83
9	261	2	---	14.42
10	39	2	---	15.14
10	43	2	---	15.02
10	47	2	---	15.43
13	103	2	---	13.73
13	104	2	---	13.48
13	105	2	---	13.84
13	106	2	---	13.61
13	107	2	---	13.26
21	1005	2	---	14.94
21	1005	2	---	14.68
21	1005	2	---	14.69
22	171	2	---	15.48
22	171	2	---	15.67
22	173	2	---	14.93
22	173	2	---	15.00
23	188	2	---	14.34
23	188	2	---	14.18
23	188	2	---	14.26
23	206	2	---	14.29
23	206	2	---	14.38
23	206	2	---	14.51
23	224	2	---	14.11
23	224	2	---	14.23
23	224	2	---	14.27
23	227	2	---	13.80
23	227	2	---	14.41
23	227	2	---	14.47

I.3.4.

Silicon Powder

Lab #	IEA Vial #	Method Code	Single- Point	Multi- Point
1	166	1	1.83	---
1	166	1	1.88	---
1	171	1	1.63	---
1	171	1	1.78	---
2	15	1	1.80	---
3	5	1	1.90	---
3	5	1	1.90	---
3	5	1	1.90	---
3	8	1	1.80	---
3	8	1	1.90	---
3	8	1	1.90	---
3	59	1	2.00	---
3	59	1	1.90	---
3	59	1	2.00	---
3	61	1	1.80	---
3	61	1	1.80	---
3	61	1	1.80	---
4	69	1	2.02	---
4	69	1	2.02	---
4	69	1	2.01	---
4	69	1	2.01	---
4	70	1	1.80	---
4	70	1	1.79	---
4	70	1	1.79	---
4	70	1	1.79	---
4	71	1	2.03	---
4	71	1	2.02	---
4	71	1	2.00	---
4	71	1	2.01	---
5	20	1	1.70	---
5	20	1	1.70	---
5	56	1	1.90	---
5	56	1	1.90	---
8	237	1	2.33	---
8	237	1	2.30	---
8	237	1	2.32	---
8	238	1	2.36	---
8	238	1	2.32	---
8	238	1	2.34	---
15	1016	1	1.50	---
16	74	1	1.99	---
16	74	1	1.96	---

Silicon Powder

Lab #	IEA Vial #	Method Code	Single- Point	Multi- Point
16	74	1	1.96	---
16	74	1	1.96	---
16	74	1	1.96	---
17	40	1	1.99	---
17	40	1	1.99	---
17	40	1	2.00	---
17	40	1	1.99	---
17	40	1	2.00	---
17	40	1	1.99	---
17	40	1	2.00	---
17	40	1	2.00	---
17	40	1	2.00	---
17	41	1	2.05	---
17	41	1	2.04	---
17	41	1	2.05	---
17	41	1	2.04	---
17	42	1	2.01	---
17	42	1	2.02	---
17	42	1	1.99	---
17	42	1	1.98	---
17	42	1	1.99	---
17	43	1	1.96	---
17	43	1	1.96	---
17	43	1	1.97	---
17	43	1	1.99	---
17	43	1	1.99	---
17	44	1	2.03	---
17	44	1	2.01	---
17	44	1	2.04	---
17	44	1	2.04	---
17	218	1	1.98	---
17	218	1	1.98	---
17	218	1	1.97	---
17	218	1	1.97	---
17	219	1	2.00	---
17	219	1	1.99	---
17	219	1	2.00	---
17	219	1	2.00	---
18	38	1	1.90	---
18	38	1	1.99	---
18	38	1	1.99	---
18	38	1	1.85	---
18	38	1	1.87	---

Silicon Powder

Lab #	IEA Vial #	Method Code	Single-Point	Multi-Point
18	38	1	1.84	---
18	38	1	1.92	---
18	38	1	1.93	---
18	38	1	1.90	---
20	35	1	2.25	---
1	166	2	---	1.98
1	166	2	---	2.02
1	171	2	---	1.78
1	171	2	---	1.95
6	81	2	---	1.71
9	201	2	---	2.18
9	201	2	---	2.16
9	201	2	---	2.19
9	242	2	---	2.20
9	242	2	---	2.57
9	242	2	---	2.21
9	243	2	---	2.19
9	243	2	---	2.16
9	243	2	---	2.61
22	95	2	---	1.98
22	95	2	---	1.98
22	95	2	---	4.67
22	95	2	---	4.25
22	96	2	---	1.95
22	96	2	---	1.95
22	96	2	---	5.32
22	96	2	---	4.50
23	105	2	---	1.90
23	105	2	---	1.98
23	105	2	---	2.04
23	106	2	---	1.93
23	106	2	---	2.01
23	106	2	---	1.99
23	703	2	---	2.03
23	703	2	---	2.00
23	703	2	---	2.06
23	1027	2	---	1.78
23	1027	2	---	1.97
23	1027	2	---	2.00

I.3.5.

Yttria-Zirconia Powder

Lab #	IEA Vial #	Method Code	Single- Point	Multi- Point
1	496	1	17.04	---
1	496	1	16.93	---
2	9	1	15.40	---
2	OB017	1	15.70	---
4	8	1	18.90	---
4	8	1	18.90	---
4	8	1	19.20	---
4	8	1	18.90	---
4	8	1	18.90	---
4	8	1	19.20	---
4	583	1	18.90	---
4	583	1	19.30	---
4	583	1	19.50	---
4	583	1	19.40	---
4	583	1	18.90	---
4	583	1	19.30	---
4	583	1	19.50	---
4	583	1	19.40	---
4	622	1	18.70	---
4	622	1	18.80	---
4	622	1	19.00	---
4	622	1	19.00	---
4	622	1	19.00	---
4	622	1	18.70	---
4	622	1	18.80	---
4	622	1	19.00	---
4	622	1	19.00	---
4	622	1	19.00	---
6	661	1	17.12	---
11	162	1	14.10	---
11	ACO15	1	16.30	---
14	430	1	17.00	---
14	603	1	16.50	---
14	603	1	16.10	---
15	OE014	1	15.80	---
16	94	1	17.20	---
16	94	1	17.40	---
16	94	1	18.80	---
16	94	1	19.00	---
16	239	1	17.40	---
16	239	1	18.10	---
16	325	1	17.40	---

Yttria-Zirconia Powder

Lab #	IEA Vial #	Method Code	Single- Point	Multi- Point
16	325	1	18.10	---
16	325	1	18.90	---
16	325	1	19.00	---
16	657	1	17.70	---
16	657	1	18.00	---
17	59	1	16.36	---
17	59	1	16.57	---
17	59	1	16.23	---
17	59	1	16.28	---
17	76	1	16.21	---
17	76	1	16.31	---
17	380	1	16.05	---
17	380	1	16.05	---
17	380	1	16.15	---
17	380	1	16.31	---
17	510	1	16.26	---
17	510	1	16.15	---
17	510	1	16.21	---
17	510	1	16.26	---
17	592	1	16.99	---
17	592	1	17.05	---
17	592	1	16.99	---
17	592	1	16.90	---
18	48	1	17.60	---
18	48	1	18.40	---
18	152	1	18.31	---
18	152	1	18.89	---
18	549	1	18.05	---
18	549	1	18.47	---
18	OR012	1	18.90	---
18	OR012	1	19.57	---
18	OR012	1	19.72	---
18	OR012	1	19.42	---
18	OR012	1	18.83	---
18	OR012	1	18.61	---
18	OR012	1	19.45	---
18	OR012	1	19.80	---
18	OR012	1	19.48	---
18	OR012	1	19.34	---
18	OR012	1	19.40	---
20	109	1	17.10	---
20	169	1	17.16	---

Yttria-Zirconia Powder

Lab #	IEA Vial #	Method Code	Single- Point	Multi- Point
20	214	1	16.90	---
24	AF017	1	15.30	---
24	OF018	1	16.50	---
24	OV018	1	15.60	---
1	496	2	---	17.67
1	496	2	---	17.53
6	661	2	---	17.62
8	153	2	---	19.09
8	217	2	---	18.61
8	601	2	---	18.64
9	210	2	---	18.98
9	254	2	---	18.99
9	376	2	---	18.91
14	16	2	---	18.20
14	16	2	---	18.40
19	31	2	---	18.28
19	82	2	---	18.12
19	224	2	---	18.17
19	297	2	---	18.03
19	396	2	---	18.14
19	428	2	---	18.44
19	582	2	---	18.09
19	593	2	---	18.45
19	OC013	2	---	18.39
22	269	2	---	19.68
22	269	2	---	19.82
22	269	2	---	18.30
22	269	2	---	19.10
22	269	2	---	19.90
22	279	2	---	18.33
22	279	2	---	18.77
22	279	2	---	18.70
22	279	2	---	18.30
22	279	2	---	19.40
22	279	2	---	18.88

APPENDIX II- Particle Size and Morphology

The methods used for particle size measurement and their code numbers are given in Table II.1.

Table II.1 Methods for Particle Size and Particle Size
Distribution Measurement

<u>Method</u>	<u>Code</u>
Electrical Sensing Zone	7
Gravitational Sedimentation (Sedigraph)	9
Centrifugal Sedimentation	10
Light-Scattering, Fraunhofer-Mie Type	13
Light-Scattering, Photon Correlation	15
Scanning Electron Microscopy	
Waddel Diameter	17
Max. Particle Diameter	18

Table II.2 Particle Size Data Organized by Powder and Measurement Method

II.2.1

Silicon Nitride Reference Powder

Gravitational Sedimentation (Sedigraph)

Lab #	IEA Vial #	Method Code	Expt. Mean μm	D_{90} , μm	D_{10} , μm	$\frac{D_{90}/D_{10}}{D_{50}}$
1	493	9	0.96	5.00	0.26	4.94
1	493	9	0.98	5.10	0.28	4.92
1	603	9	1.08	7.20	0.31	6.38
1	603	9	1.10	7.30	0.31	6.35
1	700	9	1.12	7.00	0.34	5.95
1	700	9	1.00	6.20	0.28	5.92
1	1109	9	0.88	4.10	0.25	4.38
1	1358	9	0.92	5.00	0.25	5.16
1	1423	9	0.96	7.70	0.24	7.77
1	1423	9	0.94	5.10	0.27	5.14
1	1423	9	0.87	4.90	0.24	5.36
3	542	9	1.30	4.00	0.67	2.56
3	543	9	1.35	7.30	0.62	4.95
3	1362	9	1.40	4.00	0.65	2.39
3	1369	9	1.25	3.20	0.61	2.07
4	1007	9	0.85	3.25	0.36	3.40
4	1007	9	0.77	2.25	0.37	2.44
4	1007	9	0.77	2.10	0.37	2.25
4	1007	9	0.86	4.10	0.36	4.35
4	1007	9	0.79	2.60	0.36	2.84
4	1007	9	0.79	2.60	0.36	2.84
4	1007	9	0.59	2.00	0.24	2.98
4	1347	9	0.76	3.00	0.27	3.59
4	1347	9	0.69	2.50	0.27	3.23
4	1347	9	0.42	1.70	---	---
4	1347	9	0.64	1.90	---	---
4	1347	9	0.50	1.70	---	---
4	1353	9	0.58	2.70	---	---
4	1353	9	0.57	2.25	---	---
6	449	9	0.80	2.80	0.17	3.29
6	1427	9	0.95	5.00	0.35	4.89
9	159	9	1.14	5.20	0.34	4.26
9	159	9	1.20	6.20	0.37	4.86
9	697	9	1.10	4.85	0.35	4.10
9	697	9	1.12	4.90	0.34	4.07
9	1173	9	1.18	5.90	0.34	4.71
9	1173	9	1.18	6.00	0.34	4.80
9	1752	9	1.15	5.80	0.34	4.75
9	1752	9	1.10	5.40	0.34	4.60

Silicon Nitride Reference Powder

Gravity Sedimentation (Sedigraph)

Lab #	IEA Vial #	Method Code	Expt. Mean μm	D_{90} , μm	D_{10} , μm	D_{90}/D_{10} D ₅₀
15	9013	9	1.00	5.00	0.30	4.70
16	720	9	0.78	---	---	---
16	9025	9	0.77	---	---	---
17	225	9	0.82	3.25	0.33	3.56
17	761	9	0.68	---	---	---
17	1111	9	0.90	3.50	0.29	3.57
17	1538	9	0.70	3.05	0.22	4.04
17	1538	9	0.75	3.45	0.21	4.32
17	1538	9	0.72	3.15	0.20	4.10
18	161	9	0.80	4.90	0.23	5.84
23	28	9	0.88	3.75	0.25	3.98
23	28	9	0.79	2.97	0.27	3.42
23	28	9	0.90	4.00	0.26	4.15
23	256	9	0.69	3.40	---	---
23	256	9	0.63	3.10	---	---
23	360	9	0.74	4.30	---	---
23	360	9	0.70	3.25	---	---
23	362	9	0.79	4.50	---	---
23	362	9	1.07	4.20	0.25	3.69
23	362	9	0.76	4.20	0.21	5.25
23	362	9	0.76	3.50	0.19	4.36
23	468	9	0.92	6.40	0.36	6.57
23	468	9	0.88	3.97	0.32	4.15
23	468	9	0.85	2.80	0.32	2.92
23	468	9	0.83	3.00	0.31	3.24
23	469	9	0.95	3.57	0.31	3.43
23	469	9	0.97	3.51	0.29	3.32
23	544	9	0.71	3.60	---	---
23	544	9	0.64	3.10	---	---
23	544	9	0.61	3.10	---	---
23	544	9	0.75	3.60	0.19	4.55
23	544	9	0.74	4.00	---	---
23	544	9	0.66	2.90	---	---
23	654	9	0.72	4.10	---	---
23	654	9	0.70	3.50	---	---
23	1064	9	0.86	3.68	0.26	3.98
23	1064	9	0.81	2.61	0.24	2.92
23	1064	9	0.80	2.56	0.26	2.88
23	1064	9	0.84	3.02	0.27	3.28
23	1064	9	0.62	1.70	0.23	2.37
23	1064	9	0.80	3.10	0.26	3.55
23	1169	9	0.70	3.15	0.20	4.21

Silicon Nitride Reference Powder

Lab #	IEA Vial #	Method Code	Expt. Mean μm	D ₉₀ , μm	D ₁₀ , μm	$\frac{D_{90}/D_{10}}{D_{50}}$
23	1169	9	0.79	3.85	---	---
23	1270	9	0.70	4.10	---	---
23	1270	9	0.70	3.80	---	---
23	1373	9	0.81	4.20	0.22	4.91
23	1373	9	0.79	3.50	0.18	4.20
23	1584	9	0.87	3.15	0.28	3.30
23	1584	9	0.74	2.27	0.23	2.76
1	1109	10	1.01	---	---	---
1	1109	10	1.06	---	---	---
1	1109	10	1.14	---	---	---
1	1358	10	0.91	---	---	---
1	1358	10	1.02	---	---	---
1	1358	10	1.09	---	---	---
1	1423	10	0.97	---	---	---
1	1423	10	1.00	---	---	---
1	1423	10	1.00	---	---	---
5	1741	10	0.68	4.00	0.29	5.46
5	1848	10	0.70	5.00	0.26	6.77
5	1852	10	0.67	3.80	0.30	5.22
11	9003	10	0.57	2.90	0.20	4.74
13	56	10	1.32	---	---	---
13	56	10	1.42	---	---	---
13	253	10	1.37	---	---	---
13	253	10	1.37	---	---	---
13	375	10	1.67	---	---	---
13	375	10	1.64	---	---	---
13	400	10	1.48	---	---	---
13	400	10	1.61	---	---	---
13	624	10	1.44	---	---	---
13	624	10	1.21	---	---	---
13	1016	10	1.25	---	---	---
13	1016	10	1.31	---	---	---
13	1113	10	1.39	---	---	---
13	1113	10	1.46	---	---	---
13	1281	10	1.36	---	---	---
13	1281	10	1.35	---	---	---
13	1315	10	1.39	---	---	---
13	1315	10	1.38	---	---	---
13	1880	10	1.36	---	---	---
13	1880	10	1.36	---	---	---
14	96	10	0.98	4.30	0.29	4.09
19	1171	10	1.51	---	---	---
7	1576	13	1.35	---	---	---

Silicon Nitride Reference Powder

Lab #	IEA Vial #	Method Code	Expt. Mean μm	D_{90} , μm	D_{10} , μm	D_{90}/D_{10} D ₅₀
7	1576	13	1.34	---	---	---
7	1576	13	1.33	---	---	---
7	1576	13	1.35	---	---	---
7	1576	13	1.35	---	---	---
8	984	13	1.19	6.06	0.42	4.74
8	984	13	1.24	7.33	0.42	5.57
8	984	13	1.19	6.19	0.43	4.84
8	984	13	1.19	6.38	0.42	5.01
8	984	13	1.18	5.58	0.41	4.38
8	984	13	1.17	5.72	0.41	4.54
8	984	13	1.25	6.44	0.41	4.82
8	984	13	1.13	4.70	0.40	3.81
8	1478	13	1.15	5.16	0.40	4.14
8	1478	13	0.98	3.45	0.41	3.10
8	1478	13	1.07	5.23	0.40	4.51
8	1478	13	1.10	5.27	0.40	4.43
8	1478	13	1.15	5.06	0.41	4.04
8	1478	13	1.10	4.74	0.40	3.95
8	1478	13	1.15	5.21	0.40	4.18
8	1478	13	1.14	5.46	0.41	4.43
8	1478	13	1.13	4.99	0.42	4.04
8	1478	13	1.15	5.16	0.40	4.14
8	1478	13	0.98	3.45	0.41	3.10
8	1478	13	1.11	4.42	0.40	3.62
8	1478	13	1.07	5.23	0.40	4.51
8	1478	13	1.10	5.27	0.40	4.43
8	1478	13	0.99	3.51	0.40	3.14
8	1478	13	1.15	5.06	0.41	4.04
8	1478	13	1.10	4.74	0.40	3.95
8	1478	13	1.12	5.02	0.41	4.12
8	1478	13	1.15	5.21	0.40	4.18
8	1478	13	1.14	5.46	0.41	4.43
9	159	13	1.23	6.40	0.36	4.91
9	159	13	1.22	6.40	0.36	4.95
9	159	13	1.31	7.27	0.36	5.27
9	159	13	1.24	6.68	0.36	5.10
9	159	13	1.26	6.49	0.36	4.87
9	159	13	1.27	6.53	0.35	4.87
9	697	13	1.28	7.24	0.36	5.38
9	697	13	1.20	6.31	0.36	4.96
9	697	13	1.29	7.35	0.36	5.42
9	697	13	1.25	6.68	0.36	5.06

Silicon Nitride Reference Powder

Lab #	IEA Vial #	Method Code	Expt. Mean μm	D_{90} , μm	D_{10} , μm	$\frac{D_{90}/D_{10}}{D_{50}}$
9	697	13	1.26	6.42	0.36	4.81
9	697	13	1.22	5.78	0.36	4.44
9	1173	13	1.26	6.98	0.36	5.25
9	1173	13	1.26	7.23	0.37	5.44
9	1173	13	1.28	6.88	0.36	5.09
9	1173	13	1.29	7.32	0.36	5.40
9	1173	13	1.35	7.31	0.36	5.15
9	1173	13	1.22	5.67	0.35	4.36
9	1752	13	1.14	5.44	0.34	4.47
9	1752	13	1.18	5.78	0.36	4.59
9	1752	13	1.17	5.70	0.35	4.57
9	1752	13	1.17	5.73	0.36	4.59
9	1752	13	1.18	5.90	0.36	4.69
9	1752	13	1.20	5.64	0.36	4.40
14	1686	13	0.75	2.00	0.31	2.25
14	1686	13	0.77	---	---	---
20	198	13	1.52	10.88	0.39	6.90
20	198	13	1.09	5.15	0.37	4.39
20	637	13	1.37	8.68	0.38	6.06
20	652	13	1.34	8.34	0.39	5.93
20	1241	13	1.45	11.52	0.40	7.67
20	1394	13	1.36	7.99	0.39	5.59
20	1695	13	1.31	7.83	0.39	5.68
21	9005	13	0.79	3.20	0.28	3.70
22	148	13	0.75	1.91	0.31	2.13
22	148	13	0.73	1.86	0.31	2.12
22	148	13	0.73	1.87	0.31	2.14
22	148	13	0.72	1.86	0.32	2.14
22	148	13	0.74	1.89	0.31	2.14
22	148	13	0.77	2.01	0.31	2.21
22	148	13	0.73	2.16	0.31	2.53
22	148	13	0.74	2.05	0.31	2.35
22	148	13	0.73	2.20	0.31	2.59
22	148	13	0.73	2.29	0.31	2.71
22	148	13	0.74	2.29	0.32	2.66
22	148	13	0.74	2.31	0.32	2.69
22	630	13	0.89	2.46	0.34	2.38
22	630	13	0.87	2.37	0.35	2.32
22	630	13	0.88	2.58	0.35	2.53
22	630	13	0.89	2.46	0.35	2.37
22	630	13	0.88	2.51	0.34	2.47
22	630	13	0.88	2.41	0.34	2.35

Silicon Nitride Reference Powder

Lab #	IEA Vial #	Method Code	Expt. Mean μm	D_{90} , μm	D_{10} , μm	$\frac{D_{90}/D_{10}}{D_{50}}$
22	630	13	0.84	2.58	0.34	2.67
22	630	13	0.86	2.55	0.34	2.57
22	630	13	0.84	2.55	0.35	2.62
22	630	13	0.84	2.55	0.35	2.62
22	630	13	0.83	2.55	0.35	2.65
22	630	13	0.86	2.43	0.35	2.42
22	630	13	0.88	2.31	0.35	2.23
22	630	13	0.90	2.30	0.34	2.18
22	630	13	0.86	2.49	0.36	2.48
22	630	13	0.87	2.31	0.35	2.25
22	630	13	0.90	2.27	0.33	2.16
22	630	13	0.89	2.34	0.35	2.24
22	630	13	0.74	2.06	0.32	2.35
22	630	13	0.75	2.18	0.32	2.48
22	630	13	0.75	2.20	0.33	2.49
22	630	13	0.76	2.16	0.32	2.42
22	630	13	0.75	2.16	0.32	2.45
22	630	13	0.74	2.05	0.31	2.35
24	244	13	0.40	2.90	0.10	7.00
24	667	13	0.85	9.60	0.24	11.01
25	281	13	0.80	2.91	0.33	3.23
25	289	13	0.77	2.78	0.32	3.19
25	1226	13	0.73	2.61	0.32	3.14

II.2.2.

Silicon Nitride Test Powder

Lab #	IEA Vial #	Method Code	Expt. Mean μm	D ₉₀ , μm	D ₁₀ , μm	$\frac{D_{90}/D_{10}}{D_{50}}$
1	694	9	0.55	0.91	0.33	1.05
1	694	9	0.58	0.98	0.36	1.08
3	213	9	0.69	1.15	0.43	1.04
3	1302	9	0.69	1.10	0.41	1.00
4	544	9	0.48	0.71	0.28	0.90
4	544	9	0.48	0.71	0.28	0.90
4	950	9	0.51	0.74	0.32	0.83
4	950	9	0.49	0.70	0.27	0.88
4	1375	9	0.53	0.84	0.30	1.02
6	306	9	0.64	1.03	0.38	1.02
9	200	9	0.62	1.05	0.32	1.18
9	200	9	0.71	1.10	0.37	1.03
9	1044	9	0.70	1.10	0.40	1.00
9	1044	9	0.72	1.09	0.39	0.97
9	1152	9	0.71	1.10	0.38	1.01
9	1152	9	0.64	1.06	0.32	1.16
9	1156	9	0.71	1.10	0.40	0.99
9	1156	9	0.69	1.12	0.35	1.12
9	LSM23	9	0.70	1.08	0.36	1.03
9	LSM23	9	0.72	1.10	0.44	0.92
15	LSM09	9	0.42	0.90	0.33	1.36
16	196	9	0.54	---	---	---
16	LSM0I	9	0.42	---	---	---
17	181	9	0.64	0.97	0.42	0.86
17	938	9	0.59	0.90	0.39	0.86
17	938	9	0.57	0.87	0.37	0.88
17	1314	9	0.54	0.82	0.36	0.85
17	1463	9	0.67	1.04	0.38	0.99
17	1463	9	0.64	1.00	0.37	0.98
18	626	9	0.27	0.64	---	---
23	774	9	0.50	1.00	0.28	1.44
23	774	9	0.43	0.82	0.20	1.44
23	1165	9	0.42	0.71	0.23	1.14
23	1166	9	0.44	0.78	0.26	1.18
23	1187	9	0.40	0.72	0.22	1.25
23	1187	9	0.43	0.96	0.22	1.72
23	1210	9	0.35	0.54	0.19	1.00
23	1210	9	0.46	0.78	0.27	1.11
23	1221	9	0.45	0.86	0.24	1.38
23	1344	9	0.41	0.69	0.22	1.15
23	1441	9	0.42	0.69	0.25	1.05

Silicon Nitride Test Powder

Lab #	IEA Vial #	Method Code	Expt. Mean μm	D ₉₀ , μm	D ₁₀ , μm	$\frac{D_{90}/D_{10}}{D_{50}}$
1	694	10	0.77	---	---	---
1	694	10	0.78	---	---	---
5	163	10	0.53	1.18	0.25	1.75
5	167	10	0.56	1.89	0.24	2.95
5	871	10	0.53	1.14	0.25	1.68
5	871	10	0.53	1.20	0.25	1.79
14	1178	10	0.56	1.20	0.27	1.66
14	1178	10	0.55	1.22	0.27	1.73
14	1178	10	0.66	1.81	0.34	2.23
19	101	10	1.10	---	---	---
19	101	10	0.89	---	---	---
19	217	10	0.89	---	---	---
19	621	10	0.93	---	---	---
19	621	10	0.85	---	---	---
19	959	10	0.83	---	---	---
19	1061	10	0.77	---	---	---
19	1324	10	0.84	---	---	---
19	1470	10	0.93	---	---	---
19	1470	10	0.78	---	---	---
7	564	13	0.63	---	---	---
7	564	13	0.63	---	---	---
7	564	13	0.62	---	---	---
7	564	13	0.62	---	---	---
7	564	13	0.62	---	---	---
7	976	13	0.62	---	---	---
7	976	13	0.61	---	---	---
7	976	13	0.63	---	---	---
7	976	13	0.63	---	---	---
7	976	13	0.62	---	---	---
8	223	13	0.71	1.50	0.37	1.59
8	223	13	0.71	1.52	0.37	1.62
8	223	13	0.71	1.51	0.37	1.61
8	223	13	0.69	1.48	0.37	1.61
8	223	13	0.70	1.51	0.37	1.63
8	223	13	0.70	1.50	0.37	1.61
8	223	13	0.66	1.41	0.36	1.59
8	223	13	0.66	1.39	0.36	1.56
8	223	13	0.66	1.38	0.36	1.55
8	223	13	0.65	1.36	0.35	1.55
8	223	13	0.65	1.39	0.35	1.60
8	223	13	0.65	1.37	0.36	1.55

Silicon Nitride Test Powder

Lab #	IEA Vial #	Method Code	Expt. Mean μm	D_{90} , μm	D_{10} , μm	D_{90}/D_{10} D ₅₀
8	353	13	0.71	1.53	0.37	1.63
8	353	13	0.70	1.52	0.37	1.64
8	353	13	0.72	1.57	0.37	1.67
8	353	13	0.69	1.45	0.36	1.58
8	353	13	0.69	1.47	0.37	1.59
8	353	13	0.70	1.53	0.37	1.66
8	353	13	0.68	1.46	0.36	1.62
8	353	13	0.68	1.46	0.36	1.62
8	353	13	0.68	1.47	0.36	1.63
8	353	13	0.67	1.43	0.36	1.60
8	353	13	0.67	1.43	0.36	1.60
8	353	13	0.67	1.41	0.36	1.57
8	1417	13	0.69	1.43	0.37	1.54
8	1417	13	0.69	1.47	0.37	1.59
8	1417	13	0.67	1.44	0.36	1.61
8	1417	13	0.67	1.45	0.37	1.61
8	1417	13	0.68	1.44	0.37	1.57
8	1417	13	0.66	1.42	0.36	1.61
8	1417	13	0.67	1.46	0.36	1.64
8	1417	13	0.67	1.45	0.36	1.63
8	1417	13	0.65	1.39	0.36	1.58
8	1417	13	0.66	1.39	0.36	1.56
8	1417	13	0.66	1.41	0.36	1.59
9	200	13	0.61	1.28	0.30	1.61
9	200	13	0.61	1.27	0.30	1.59
9	200	13	0.61	1.30	0.30	1.64
9	200	13	0.61	1.26	0.30	1.57
9	200	13	0.61	1.25	0.30	1.56
9	200	13	0.61	1.24	0.30	1.54
9	200	13	0.61	1.34	0.30	1.70
9	200	13	0.61	1.37	0.30	1.75
9	200	13	0.61	1.38	0.31	1.75
9	200	13	0.61	1.37	0.30	1.75
9	200	13	0.61	1.40	0.30	1.80
9	200	13	0.61	1.35	0.30	1.72
9	1044	13	0.61	1.31	0.29	1.67
9	1044	13	0.60	1.26	0.29	1.62
9	1044	13	0.61	1.24	0.30	1.54
9	1044	13	0.61	1.29	0.30	1.62
9	1044	13	0.61	1.28	0.30	1.61
9	1044	13	0.61	1.29	0.30	1.62
9	1044	13	0.61	1.34	0.31	1.69

Silicon Nitride Test Powder

Lab #	IEA Vial #	Method Code	Expt. Mean μm	D ₉₀ , μm	D ₁₀ , μm	$\frac{D_{90}/D_{10}}{D_{50}}$
9	1044	13	0.61	1.38	0.31	1.75
9	1044	13	0.61	1.35	0.30	1.72
9	1044	13	0.61	1.36	0.30	1.74
9	1044	13	0.61	1.37	0.31	1.74
9	1044	13	0.62	1.39	0.31	1.74
9	1152	13	0.60	1.23	0.29	1.57
9	1152	13	0.60	1.25	0.30	1.58
9	1152	13	0.62	1.41	0.30	1.79
9	1152	13	0.60	1.25	0.30	1.58
9	1152	13	0.60	1.24	0.30	1.57
9	1152	13	0.60	1.25	0.29	1.60
9	1156	13	0.60	1.24	0.29	1.58
9	1156	13	0.60	1.22	0.29	1.55
9	1156	13	0.62	1.49	0.30	1.92
9	1156	13	0.60	1.18	0.30	1.47
9	1156	13	0.60	1.23	0.29	1.57
9	1156	13	0.60	1.23	0.30	1.55
9	1156	13	0.62	1.37	0.31	1.71
9	1156	13	0.62	1.36	0.30	1.71
9	1156	13	0.62	1.43	0.32	1.79
9	1156	13	0.62	1.42	0.31	1.79
9	1156	13	0.62	1.41	0.31	1.77
9	1156	13	0.62	1.36	0.31	1.69
9	LSM23	13	0.63	1.37	0.31	1.68
9	LSM23	13	0.62	1.30	0.32	1.58
9	LSM23	13	0.62	1.35	0.30	1.69
9	LSM23	13	0.62	1.32	0.32	1.61
9	LSM23	13	0.63	1.26	0.31	1.51
9	LSM23	13	0.62	1.34	0.31	1.66
9	LSM23	13	0.61	1.40	0.31	1.79
9	LSM23	13	0.61	1.29	0.30	1.62
9	LSM23	13	0.61	1.40	0.30	1.80
9	LSM23	13	0.61	1.38	0.30	1.77
9	LSM23	13	0.60	1.40	0.28	1.87
9	LSM23	13	0.62	1.40	0.30	1.77
14	222	13	0.53	1.50	0.17	2.51
14	222	13	0.57	1.25	0.23	1.79
14	960	13	0.55	1.60	0.22	2.51
14	960	13	0.52	1.33	0.22	2.13
20	193	13	0.58	1.10	0.32	1.34
20	538	13	0.58	1.09	0.32	1.33
20	850	13	0.58	1.11	0.32	1.36

Silicon Nitride Test Powder

Lab #	IEA Vial #	Method Code	Expt. Mean μm	D_{90} , μm	D_{10} , μm	$\frac{D_{90}/D_{10}}{D_{50}}$
22	94	13	0.58	1.24	0.31	1.60
22	94	13	0.57	1.10	0.31	1.39
22	94	13	0.58	1.21	0.31	1.55
22	94	13	0.57	1.18	0.31	1.53
22	94	13	0.56	1.02	0.30	1.29
22	94	13	0.58	1.24	0.31	1.60
22	94	13	0.58	1.20	0.31	1.53
22	94	13	0.56	1.06	0.30	1.36
22	94	13	0.56	1.08	0.30	1.39
22	94	13	0.56	1.05	0.31	1.32
22	94	13	0.57	1.24	0.30	1.65
22	94	13	0.56	1.19	0.30	1.59
22	94	13	0.60	1.26	0.32	1.57
22	94	13	0.60	1.32	0.32	1.67
22	94	13	0.59	1.23	0.31	1.56
22	94	13	0.58	1.13	0.31	1.41
22	94	13	0.59	1.17	0.31	1.46
22	94	13	0.59	1.29	0.32	1.64
22	502	13	0.58	1.13	0.31	1.41
22	502	13	0.58	1.20	0.31	1.53
22	502	13	0.57	1.11	0.31	1.40
22	502	13	0.57	1.05	0.31	1.30
22	502	13	0.57	1.03	0.31	1.26
22	502	13	0.57	1.10	0.31	1.39
22	502	13	0.57	0.99	0.32	1.18
22	502	13	0.56	0.99	0.30	1.23
22	502	13	0.56	1.08	0.30	1.39
22	502	13	0.56	1.00	0.30	1.25
22	502	13	0.56	1.13	0.30	1.48
22	502	13	0.56	1.01	0.30	1.27
24	298	13	0.73	1.51	0.34	1.61
24	298	13	0.77	1.72	0.41	1.70
24	895	13	0.75	2.01	0.34	2.23
24	895	13	0.69	1.92	0.32	2.32
25	432	13	0.54	1.00	0.30	1.30
25	432	13	0.55	0.99	0.30	1.25
25	758	13	0.55	1.01	0.30	1.29

II.2.3.

Silicon Carbide Powder

Lab #	IEA Vial #	Method Code	Expt. Mean μm	D ₉₀ , μm	D ₁₀ , μm	$\frac{D_{90}/D_{10}}{D_{50}}$
1	237	9	0.75	1.20	0.53	0.89
1	237	9	0.76	1.20	0.52	0.89
1	243	9	0.76	1.50	0.53	1.28
3	4	9	4.00	7.20	4.10	0.78
3	4	9	4.80	6.50	3.90	0.54
3	4	9	4.80	6.60	3.90	0.56
3	4	9	4.20	6.00	2.80	0.76
3	7	9	4.50	6.60	3.90	0.60
3	7	9	4.40	6.00	3.50	0.57
3	7	9	4.00	5.80	2.90	0.73
3	9	9	4.60	6.40	3.80	0.57
3	9	9	4.40	5.90	3.70	0.50
3	9	9	4.20	6.40	3.50	0.69
3	11	9	5.00	6.70	4.40	0.46
3	11	9	4.40	6.20	3.90	0.52
3	11	9	4.10	6.00	3.80	0.54
4	69	9	0.65	0.95	0.46	0.75
4	69	9	0.57	0.88	0.43	0.79
4	69	9	0.49	0.80	0.36	0.90
6	99	9	0.82	1.30	0.62	0.83
9	255	9	0.59	1.12	0.20	1.55
9	255	9	0.58	1.15	0.21	1.62
9	256	9	0.58	1.10	0.20	1.54
9	256	9	0.58	1.15	0.20	1.63
9	261	9	0.58	1.25	0.20	1.80
9	261	9	0.53	1.05	0.20	1.60
9	261	9	0.52	1.05	0.19	1.65
10	1025	9	0.90	1.50	---	---
15	1012	9	0.55	1.00	0.20	1.45
16	82	9	0.53	1.00	0.19	1.53
16	LSM1013	9	0.46	0.85	0.20	1.41
23	187	9	0.70	1.20	0.51	0.99
23	187	9	0.74	1.55	0.50	1.42
23	190	9	0.55	0.98	0.35	1.14
23	190	9	0.71	1.55	0.35	1.69
23	201	9	0.65	1.45	0.45	1.54
23	201	9	0.63	1.35	0.45	1.43
23	201	9	0.60	1.05	0.41	1.07
23	201	9	0.59	1.05	0.39	1.12
23	202	9	1.03	2.15	0.55	1.55
23	202	9	0.70	1.61	0.46	1.64
23	225	9	0.92	2.00	0.51	1.62

Silicon Carbide Powder

Lab #	IEA Vial #	Method Code	Expt. Mean μm	D ₉₀ , μm	D ₁₀ , μm	D ₉₀ -D ₁₀ D ₅₀
23	225	9	0.99	1.78	0.55	1.24
23	225	9	0.62	1.32	0.41	1.47
23	228	9	0.76	1.45	0.51	1.24
23	228	9	0.75	1.35	0.55	1.07
23	228	9	0.72	1.35	0.50	1.18
23	229	9	0.74	1.45	0.51	1.27
23	229	9	0.67	1.25	0.48	1.15
23	234	9	0.69	1.40	0.45	1.38
23	234	9	0.80	1.50	0.49	1.26
23	234	9	0.98	1.80	0.50	1.33
1	236	10	0.68	1.88	0.40	2.18
1	240	10	0.71	1.50	0.45	1.48
1	243	10	0.83	2.06	0.45	1.94
1	243	10	0.64	1.80	0.32	2.31
1	243	10	0.58	1.72	0.29	2.47
1	243	10	0.70	1.76	0.38	1.97
2	13	10	0.40	0.86	0.11	1.87
13	103	10	0.63	---	---	---
13	103	10	0.65	---	---	---
13	104	10	0.71	---	---	---
13	104	10	0.70	---	---	---
13	105	10	0.66	---	---	---
13	105	10	0.67	---	---	---
13	106	10	0.67	---	---	---
13	106	10	0.65	---	---	---
13	107	10	0.65	---	---	---
13	107	10	0.68	---	---	---
7	250	13	0.44	0.67	0.29	0.86
7	250	13	0.39	0.64	0.23	1.05
7	250	13	0.44	0.67	0.29	0.86
8	268	13	0.52	1.00	0.30	1.35
8	268	13	0.52	1.00	0.30	1.35
8	268	13	0.52	0.99	0.30	1.33
8	268	13	0.51	0.99	0.30	1.35
8	268	13	0.51	0.99	0.30	1.35
8	268	13	0.52	1.01	0.30	1.37
8	268	13	0.51	0.98	0.30	1.33
8	268	13	0.51	0.99	0.29	1.37
8	268	13	0.51	0.99	0.30	1.35
8	268	13	0.51	0.99	0.30	1.35

Silicon Carbide Powder

Lab #	IEA Vial #	Method Code	Expt. Mean μm	D ₉₀ , μm	D ₁₀ , μm	D ₉₀ /D ₁₀ D ₅₀
8	268	13	0.51	0.99	0.30	1.35
8	268	13	0.51	1.00	0.29	1.39
8	271	13	0.51	0.99	0.30	1.35
8	271	13	0.51	0.99	0.30	1.35
8	271	13	0.51	0.99	0.30	1.35
8	271	13	0.51	0.98	0.30	1.33
8	271	13	0.51	0.98	0.30	1.33
8	271	13	0.51	0.98	0.29	1.35
8	271	13	0.51	0.99	0.30	1.35
8	271	13	0.51	0.98	0.30	1.33
8	271	13	0.50	0.99	0.29	1.40
8	271	13	0.51	0.98	0.30	1.33
8	271	13	0.51	0.99	0.30	1.35
8	271	13	0.52	0.99	0.30	1.33
10	39	13	0.49	0.99	0.23	1.55
10	40	13	0.50	1.52	0.23	2.58
10	43	13	0.48	1.03	0.23	1.67
10	1018	13	0.48	1.24	0.23	2.10
20	90	13	0.72	1.61	0.35	1.75
21	1005	13	0.57	---	---	---
22	171	13	0.64	1.50	0.33	1.83
22	171	13	0.63	1.41	0.29	1.78
22	171	13	0.59	1.32	0.30	1.73
22	171	13	0.58	1.42	0.30	1.93
22	171	13	0.58	1.25	0.31	1.62
22	171	13	0.57	1.17	0.30	1.53
22	171	13	0.66	1.41	0.36	1.59
22	171	13	0.61	1.24	0.35	1.46
22	171	13	0.60	1.30	0.33	1.62
22	171	13	0.60	1.20	0.34	1.43
22	171	13	0.59	1.23	0.32	1.54
22	171	13	0.59	1.23	0.33	1.53
22	171	13	0.65	1.37	0.35	1.57
22	171	13	0.59	1.00	0.35	1.10
22	171	13	0.61	1.31	0.33	1.61
22	171	13	0.59	0.98	0.35	1.07
22	171	13	0.59	1.18	0.34	1.42
22	171	13	0.57	0.98	0.33	1.14
22	171	13	0.64	1.35	0.35	1.56
22	171	13	0.60	1.23	0.33	1.50
22	171	13	0.59	1.19	0.32	1.47
22	171	13	0.58	1.11	0.32	1.36

Silicon Carbide Powder

Lab #	IEA Vial #	Method Code	Expt. Mean μm	D_{90} , μm	D_{10} , μm	$\frac{D_{90}/D_{10}}{D_{50}}$
22	171	13	0.58	1.15	0.32	1.43
22	171	13	0.58	1.14	0.32	1.41
22	174	13	0.65	1.37	0.35	1.57
22	174	13	0.61	1.22	0.34	1.44
22	174	13	0.60	1.19	0.34	1.42
22	174	13	0.59	1.19	0.34	1.44
22	174	13	0.58	1.09	0.33	1.31
22	174	13	0.58	1.16	0.33	1.43
22	174	13	0.65	1.45	0.34	1.71
22	174	13	0.60	1.24	0.35	1.48
22	174	13	0.59	1.08	0.34	1.25
22	174	13	0.59	1.22	0.32	1.53
22	174	13	0.58	1.08	0.34	1.28
22	174	13	0.57	1.09	0.33	1.33
22	174	13	0.62	1.64	0.31	2.15
22	174	13	0.56	0.91	0.34	1.02
22	174	13	0.58	1.09	0.33	1.31
22	174	13	0.57	1.18	0.31	1.53
22	174	13	0.55	1.29	0.30	1.80
22	174	13	0.62	1.41	0.33	1.74
22	174	13	0.59	1.32	0.32	1.69
22	174	13	0.57	1.15	0.31	1.47
22	174	13	0.60	1.53	0.32	2.02
22	174	13	0.55	1.13	0.30	1.51
22	174	13	0.57	0.99	0.33	1.16
25	1016	13	0.53	1.17	0.29	1.66

II.2.4.

Silicon Powder

Lab #	IEA Vial #	Method Code	Expt. Mean μm	D ₉₀ , μm	D ₁₀ , μm	$\frac{D_{90}/D_{10}}{D_{50}}$
16	74	7	5.30	9.90	1.08	1.66
16	74	7	5.20	9.60	1.07	1.64
16	74	7	5.20	9.60	1.05	1.64
16	74	7	5.40	10.00	1.08	1.65
16	74	7	5.50	10.00	1.15	1.61
16	LSM1013	7	5.30	---	---	---
1	166	9	6.70	12.50	1.75	1.60
1	166	9	6.70	12.00	1.90	1.51
1	168	9	6.80	12.80	1.85	1.61
3	5	9	5.80	10.50	2.10	1.45
3	5	9	5.80	10.50	1.90	1.48
3	5	9	5.40	9.50	1.40	1.50
3	8	9	5.90	10.80	1.95	1.50
3	8	9	5.90	10.80	1.82	1.52
3	8	9	5.90	10.60	1.65	1.52
3	59	9	6.30	10.50	2.30	1.30
3	59	9	5.70	10.50	1.70	1.54
3	59	9	5.70	10.50	1.55	1.57
3	61	9	5.80	10.40	2.10	1.43
3	61	9	5.80	10.40	1.95	1.46
4	69	9	5.40	10.30	1.05	1.71
4	69	9	5.40	10.30	1.01	1.72
4	71	9	5.70	10.50	1.35	1.61
4	71	9	5.50	10.10	1.35	1.59
4	72	9	5.90	10.80	1.55	1.57
4	72	9	5.90	10.80	1.55	1.57
6	82	9	5.80	10.07	1.55	1.47
9	201	9	6.50	11.50	2.00	1.46
9	201	9	6.60	11.50	2.50	1.37
9	242	9	6.50	11.50	2.35	1.41
9	242	9	6.70	12.00	2.00	1.49
9	243	9	6.80	12.30	2.50	1.44
9	243	9	6.60	11.50	2.30	1.39
15	1016	9	6.20	10.50	2.00	1.37
16	32	9	5.65	9.67	2.03	1.35
16	LSM1013	9	5.58	9.52	2.13	1.32
17	40	9	7.20	13.00	1.80	1.56
17	40	9	7.20	13.00	1.80	1.56
17	40	9	7.30	13.00	1.90	1.52
17	42	9	7.50	13.80	2.00	1.57
17	42	9	7.50	13.80	2.00	1.57

Silicon Powder

Lab #	IEA Vial #	Method Code	Expt. Mean μm	D ₉₀ , μm	D ₁₀ , μm	D ₉₀ /D ₁₀ D ₅₀
17	42	9	7.40	13.50	1.80	1.58
17	42	9	7.20	13.50	---	---
17	42	9	7.00	13.00	1.75	1.61
17	90	9	6.70	12.50	1.55	1.63
17	90	9	6.70	12.00	1.55	1.56
17	128	9	7.60	13.70	2.00	1.54
17	128	9	7.60	14.20	2.00	1.61
17	128	9	7.40	13.50	2.00	1.55
18	36	9	5.60	10.00	1.50	1.52
23	48	9	6.50	12.30	2.05	1.58
23	101	9	6.20	13.00	1.60	1.84
23	102	9	6.90	12.30	2.25	1.46
23	102	9	6.80	13.50	1.50	1.76
23	1022	9	6.20	12.00	1.80	1.65
23	1024	9	6.30	13.30	1.70	1.84
23	1024	9	6.30	13.00	1.70	1.79
23	1026	9	6.10	12.20	1.50	1.75
23	1032	9	6.50	13.30	2.30	1.69
23	1032	9	6.10	13.30	1.30	1.97
23	1034	9	5.70	14.20	1.35	2.25
1	166	10	4.15	7.49	1.40	1.47
1	166	10	4.29	7.06	1.95	1.19
1	166	10	4.09	7.35	1.95	1.32
2	65	10	6.60	9.80	0.43	1.42
5	1	10	5.00	11.80	0.56	2.25
5	24	10	4.90	11.70	0.56	2.27
19	227	10	4.79	---	---	---
19	228	10	5.30	---	---	---
19	229	10	4.96	---	---	---
19	230	10	4.85	---	---	---
19	231	10	5.39	---	---	---
19	232	10	6.05	---	---	---
19	233	10	4.62	---	---	---
19	244	10	5.03	---	---	---
19	1005	10	4.74	---	---	---
5	1	13	6.50	11.70	1.60	1.55
5	24	13	6.50	11.80	1.60	1.57
8	237	13	7.31	13.99	2.57	1.56
8	237	13	7.58	15.01	2.55	1.64
8	237	13	7.51	14.39	2.52	1.58

Silicon Powder

Lab #	IEA Vial #	Method Code	Expt. Mean μm	D ₉₀ , μm	D ₁₀ , μm	$\frac{D_{90}/D_{10}}{D_{50}}$
8	237	13	7.34	13.94	2.58	1.55
8	237	13	7.55	14.43	2.65	1.56
8	237	13	7.58	14.16	2.51	1.54
8	237	13	7.40	13.45	2.60	1.47
8	237	13	7.78	14.04	2.81	1.44
8	237	13	7.36	13.98	2.55	1.55
8	237	13	7.29	13.70	2.62	1.52
8	237	13	7.42	13.26	2.71	1.42
8	237	13	7.24	13.27	2.63	1.47
8	238	13	7.49	14.49	2.58	1.59
8	238	13	7.37	13.63	2.51	1.51
8	238	13	7.28	13.90	2.47	1.57
8	238	13	7.12	13.77	2.48	1.59
8	238	13	7.29	14.00	2.44	1.59
8	238	13	7.56	14.52	2.80	1.55
8	238	13	7.33	14.30	2.63	1.59
8	238	13	7.35	14.19	2.60	1.58
8	238	13	7.23	13.60	2.66	1.51
8	238	13	7.19	13.62	2.63	1.53
8	238	13	7.12	12.84	2.40	1.47
8	238	13	7.39	14.41	2.64	1.59
9	201	13	7.40	13.70	2.85	1.47
9	201	13	7.47	14.05	2.96	1.48
9	201	13	7.44	14.39	2.91	1.54
9	201	13	7.49	14.08	2.92	1.49
9	201	13	7.43	14.04	2.92	1.50
9	201	13	7.47	14.26	2.92	1.52
9	201	13	7.40	13.70	2.85	1.47
9	201	13	7.47	14.05	2.96	1.48
9	201	13	7.44	14.39	2.91	1.54
9	201	13	7.49	14.08	2.92	1.49
9	201	13	7.43	14.04	2.92	1.50
9	201	13	7.47	14.26	2.92	1.52
9	242	13	7.42	14.03	2.81	1.51
9	242	13	7.43	14.06	2.87	1.51
9	242	13	7.40	13.74	2.83	1.47
9	242	13	7.52	13.92	2.89	1.47
9	242	13	7.47	14.08	2.96	1.49
9	242	13	7.41	14.00	2.84	1.51
9	242	13	7.42	14.03	2.81	1.51
9	242	13	7.43	14.06	2.87	1.51
9	242	13	7.40	13.74	2.83	1.47

Silicon Powder

Lab #	IEA Vial #	Method Code	Expt. Mean μm	D ₉₀ , μm	D ₁₀ , μm	$\frac{D_{90}/D_{10}}{D_{50}}$
9	242	13	7.52	13.92	2.89	1.47
9	242	13	7.47	14.08	2.96	1.49
9	242	13	7.41	14.00	2.84	1.51
9	243	13	7.34	13.80	2.81	1.50
9	243	13	7.39	13.60	2.86	1.45
9	243	13	7.37	13.85	2.86	1.49
9	243	13	7.46	14.00	2.88	1.49
9	243	13	7.61	14.16	3.02	1.46
9	243	13	7.39	13.96	2.73	1.53
9	243	13	7.34	13.80	2.81	1.50
9	243	13	7.39	13.60	2.86	1.45
9	243	13	7.37	13.85	2.86	1.49
9	243	13	7.46	14.00	2.88	1.49
9	243	13	7.61	14.16	3.02	1.46
9	243	13	7.39	13.96	2.73	1.52
20	35	13	7.36	13.89	2.70	1.52
22	93	13	8.10	14.87	2.18	1.57
22	93	13	7.81	14.34	2.06	1.57
22	93	13	7.69	14.82	2.17	1.64
22	93	13	7.59	14.11	2.15	1.58
22	93	13	7.85	14.97	2.15	1.63
22	93	13	7.84	15.14	2.16	1.66
22	93	13	7.33	13.98	2.50	1.57
22	93	13	7.47	13.96	2.32	1.56
22	93	13	7.49	13.88	2.42	1.53
22	93	13	7.34	13.09	2.06	1.50
22	93	13	7.68	14.45	2.19	1.60
22	93	13	7.63	13.84	2.18	1.53
22	94	13	7.09	12.80	2.32	1.48
22	94	13	7.22	13.50	2.32	1.55
22	94	13	7.28	13.44	2.61	1.49
22	94	13	7.19	12.90	2.21	1.49
22	94	13	7.37	13.45	2.12	1.54
22	94	13	7.25	13.68	2.17	1.59
22	94	13	7.63	12.86	2.70	1.33
22	94	13	7.59	12.61	2.68	1.31
22	94	13	7.91	13.71	2.53	1.41
22	94	13	7.83	13.69	2.94	1.37
22	94	13	7.90	13.75	2.61	1.41
22	94	13	7.70	13.58	2.74	1.41

II.2.5.

Yttria-Zirconia Powder

Lab #	IEA Vial #	Method Code	Expt. Mean μm	D ₉₀ , μm	D ₁₀ , μm	$\frac{D_{90}/D_{10}}{D_{50}}$
4	313	9	0.17	3.20	---	---
4	313	9	---	2.60	---	---
4	432	9	0.15	3.00	---	---
4	432	9	0.15	3.00	---	---
4	497	9	0.15	2.70	---	---
4	497	9	---	2.20	---	---
4	497	9	0.32	4.00	---	---
4	497	9	0.28	3.50	---	---
6	470	9	0.20	3.20	---	---
15	OE014	9	0.20	4.00	---	---
17	59	9	0.24	---	---	---
17	59	9	0.18	---	---	---
17	76	9	0.18	---	---	---
17	76	9	0.17	---	---	---
17	76	9	0.16	---	---	---
17	225	9	0.24	4.00	---	---
17	225	9	0.24	3.70	---	---
17	380	9	0.19	---	---	---
17	380	9	0.14	---	---	---
17	380	9	0.17	---	---	---
17	380	9	0.15	---	---	---
17	380	9	0.18	---	---	---
17	380	9	0.20	---	---	---
17	380	9	0.20	---	---	---
17	380	9	0.21	---	---	---
17	380	9	0.19	---	---	---
17	380	9	0.19	---	---	---
17	TUBE#1	9	0.76	6.70	---	---
17	TUBE#1	9	0.40	7.20	---	---
17	TUBE#2	9	0.60	8.50	---	---
17	TUBE#3	9	0.31	6.60	0.16	20.78
18	48	9	0.15	10.50	---	---
18	101	9	0.50	11.00	---	---
18	549	9	0.15	12.00	---	---
18	OR12	9	0.15	11.00	---	---
18	OR12	9	0.17	14.00	---	---
23	46	9	0.15	---	---	---
23	46	9	0.18	---	---	---
1	731	10	1.54	---	---	---
1	731	10	1.44	---	---	---
1	731	10	1.47	---	---	---

Yttria-Zirconia Powder

Lab #	IEA Vial #	Method Code	Expt. Mean μm	D ₉₀ , μm	D ₁₀ , μm	$\frac{D_{90}/D_{10}}{D_{50}}$
1	731	10	1.62	---	---	---
1	731	10	1.54	---	---	---
1	731	10	1.38	---	---	---
11	162	10	0.56	2.90	0.18	4.86
14	462	10	1.73	7.30	0.29	4.05
19	31	10	1.50	---	---	---
19	31	10	1.50	---	---	---
19	82	10	1.34	---	---	---
19	82	10	1.43	---	---	---
19	224	10	1.59	---	---	---
19	224	10	1.54	---	---	---
19	297	10	1.40	---	---	---
19	297	10	1.47	---	---	---
19	396	10	1.41	---	---	---
19	396	10	1.21	---	---	---
19	428	10	1.58	---	---	---
19	428	10	1.62	---	---	---
19	582	10	1.69	---	---	---
19	582	10	1.61	---	---	---
19	593	10	1.50	---	---	---
19	593	10	1.49	---	---	---
19	OC013	10	1.38	---	---	---
19	OC013	10	1.34	---	---	---
11	AC015	13	2.90	9.20	0.37	3.04
14	232	13	2.20	9.50	0.40	4.14
14	562	13	2.30	9.50	0.40	3.96
14	567	13	2.30	9.80	0.40	4.09
20	109	13	4.06	11.33	0.51	2.67
20	169	13	4.17	11.80	0.50	2.71
20	214	13	4.19	11.49	0.50	2.62
22	269	13	0.62	2.60	0.15	3.95
22	269	13	0.66	2.87	0.15	4.12
22	269	13	0.72	2.55	0.15	3.33
22	269	13	0.95	2.72	0.15	2.71
22	269	13	0.82	2.50	0.15	2.87
22	269	13	0.78	2.38	0.15	2.86
22	269	13	0.83	2.96	0.15	3.39
24	AF017	13	1.50	7.70	0.13	5.05
24	OF018	13	1.80	8.60	0.14	4.70
24	OV018	13	1.40	7.60	0.13	5.34

Yttria-Zirconia Powder

Lab #	IEA Vial #	Method Code	Expt. Mean μm	D ₉₀ , μm	D ₁₀ , μm	$\frac{D_{90}/D_{10}}{D_{50}}$
9	210	15	0.21	---	---	---
9	210	15	0.22	---	---	---
9	210	15	0.20	---	---	---
9	210	15	0.21	---	---	---
9	210	15	0.19	---	---	---
9	210	15	0.20	---	---	---
9	210	15	0.19	---	---	---
9	210	15	0.19	---	---	---
9	254	15	0.20	---	---	---
9	254	15	0.19	---	---	---
9	254	15	0.20	---	---	---
9	254	15	0.19	---	---	---
9	254	15	0.19	---	---	---
9	254	15	0.19	---	---	---
9	254	15	0.19	---	---	---
9	254	15	0.19	---	---	---
9	376	15	0.22	---	---	---
9	376	15	0.22	---	---	---
9	376	15	0.21	---	---	---
9	376	15	0.22	---	---	---
9	376	15	0.19	---	---	---
9	376	15	0.19	---	---	---
9	376	15	0.19	---	---	---
9	376	15	0.18	---	---	---
23	46	15	0.18	---	---	---
23	46	15	0.18	---	---	---
23	46	15	0.18	---	---	---
23	46	15	0.19	---	---	---
23	46	15	0.18	---	---	---
23	46	15	0.18	---	---	---
23	46	15	0.18	---	---	---
23	46	15	0.18	---	---	---
23	46	15	0.18	---	---	---
23	46	15	0.18	---	---	---

Table II.3 PARTICLE MORPHOLOGY - DATA TABLES

II.3.1. Silicon Nitride Reference Powder, Number Distribution

Lab #	IEA #	Method Code	Expt. Mean μm	Geom. Std. Dev. σ_g
---	-----	-----	-----	-----
23	609	17	0.4	--
13	56	18	0.80	--
13	253	18	0.86	--
13	375	18	0.81	--
13	400	18	0.69	--
13	624	18	0.72	--
13	1016	18	0.81	--
13	1113	18	0.98	--
13	1281	18	0.76	--
13	1315	18	0.79	--
13	1880	18	0.78	--
13	LSM 9012	18	0.62	--

II.3.2. Silicon Nitride Reference Powder, Mass Distribution

Lab #	IEA #	Method Code	Expt. Mean μm	Geom. Std. Dev. σ_g
---	-----	-----	-----	-----
23	609	17	1.39	2.29
12	9011	18	1.50	--

II.3.3. Silicon Nitride Test Powder, Number Distribution

Lab #	IEA #	Method Code	Expt. Mean μm	Geom. Std. Dev. σ_g
---	-----	-----	-----	-----
23	284	17	0.5	--
23	284	17	0.4	--

II.3.4. Silicon Nitride Test Powder, Mass Distribution

Lab #	IEA #	Method Code	Expt. Mean μm	Geom. Std. Dev. σ_g
---	-----	-----	-----	-----
23	284	17	1.03	1.83
23	284	17	1.06	2.02
12	LSM 16	18	1.20	--

II.3.5. Silicon Carbide Powder, Number Distribution

Lab #	IEA #	Method Code	Expt. Mean μm	Geom. Std. Dev. σ_g
---	-----	-----	-----	-----
23	191	17	0.32	1.96
23	191	17	0.35	1.78
23	191	17	0.41	1.64

II.3.6. Silicon Carbide Powder, Mass Distribution

Lab #	IEA #	Method Code	Expt. Mean μm	Geom. Std. Dev. σ_g
---	-----	-----	-----	-----
23	191	17	0.79	1.75
23	191	17	0.83	1.61
23	191	17	0.90	1.69

II.3.7. Silicon Powder, Number Distribution

Lab #	IEA #	Method Code	Expt. Mean μm	Geom. Std. Dev. σ_g
---	-----	-----	-----	-----
23	47	17	1.16	2.29
23	47	17	1.16	2.15
23	47	17	1.16	2.33
23	47	17	1.23	2.27
23	1028	17	1.11	2.10
23	1028	17	1.06	2.26
12	1012	18	2.10	--

II.3.8. Silicon Powder, Mass Distribution

Lab #	IEA #	Method Code	Expt. Mean μm	Geom. Std. Dev. σ_g
---	-----	-----	-----	-----
23	47	17	6.65	1.93
23	47	17	6.15	1.94
23	47	17	6.53	1.98
23	47	17	7.15	1.78
23	1028	17	5.47	1.98
23	1028	17	4.89	2.04

II.3.9. Yttria-Zirconia Powder, As-Received, Number Distribution

Lab #	IEA #	Method Code	Expt. Mean μm	Std. Dev.
---	-----	-----	-----	-----
18	OR012	18	39.36	17.04
18	OR012	18	31.10	17.87
18	OR012	18	36.16	20.23
18	215	18	34.95	16.28
18	48	18	34.83	17.97
18	549	18	27.49	16.31

II.3.10. Yttria-Zirconia Powder, Dispersed, Number Distribution

Lab #	IEA #	Method Code	Expt. Mean μm	Geom. Std. Dev. σ_g
---	-----	-----	-----	-----
23	198	17	0.3	--

II.3.11. Yttria-Zirconia Powder, Dispersed, Mass Distribution

Lab #	IEA #	Method Code	Expt. Mean μm	Geom. Std. Dev. σ_g
---	-----	-----	-----	-----
23	198	17	2.27	4.39
12	30306403	18	1.50	--
12	30306403	18	1.80	--

APPENDIX III - Chemical Composition

Table III.1 lists the methods and their code numbers for identification of these methods in the following data tables.

Table III.1 Chemical Analysis Methods and Codes

<u>Method</u>	<u>Code</u>
Atomic Absorption/Emission (AA/AE)	1
Inductively Coupled Plasma (ICP)	2
Ion Chromatography	3
Specific Ion Electrode	4
Mass Spectrometry (MS)	5
Neutron Activation Analysis (NAA)	6
Optical Emission Analysis	7
Combustion	8
X-Ray Fluorescence (XRF)	12
Potentiometric Titration	13
Argentometric (Coulometric) T.	14
Kjeldahl Analysis	15
Coulometric Titration	16
Directly Coupled Plasma (DCP)	17
Gravimetric	20
Not specified	21

Table III.2 Major Chemical Component

III.2.1.

Silicon Nitride Reference Powder (SNR)

Lab #	IEA Vial #	Method Code	Si %	N %	Si (free) %
14	103	1	59.50	---	---
14	593	1	59.50	---	---
1	396	2	58.50	---	---
1	396	2	56.30	---	---
6	1677	2	58.07	---	---
15	965	2	59.38	---	---
21	634	2	59.92	---	---
21	634	2	59.65	---	---
21	634	2	58.68	---	---
21	634	2	59.43	---	---
1	373	8	---	38.33	---
1	373	8	---	38.63	---
1	1878	8	---	38.45	---
1	1878	8	---	38.67	---
1	1878	8	---	38.57	---
1	1878	8	---	38.40	---
3	525	8	---	38.30	---
3	525	8	---	38.30	---
3	527	8	---	38.20	---
3	527	8	---	38.20	---
3	535	8	---	38.20	---
3	535	8	---	38.20	---
3	538	8	---	38.20	---
3	538	8	---	38.20	---
5	1620	8	---	38.50	---
6	106	8	---	38.40	---
6	106	8	---	38.50	---
6	756	8	---	37.70	---
6	756	8	---	37.80	---
13	56	8	---	40.85	---
13	253	8	---	40.57	---
13	375	8	---	40.50	---
13	400	8	---	40.56	---
13	624	8	---	40.20	---
13	1016	8	---	40.03	---
13	1113	8	---	40.72	---
13	1281	8	---	41.32	---
13	1315	8	---	41.23	---
13	1880	8	---	41.52	---
13	9012	8	---	41.26	---

Silicon Nitride Reference Powder

Lab #	IEA Vial #	Method Code	Si %	N %	Si (free) %
14	103	8	---	38.00	---
18	577	8	---	32.10	---
18	885	8	---	37.58	---
19	1171	8	---	37.72	---
21	634	8	---	38.30	---
21	634	8	---	38.10	---
21	634	8	---	38.20	---
24	244	8	---	40.50	---
24	1704	8	---	39.30	---
24	9005	8	---	38.30	---
5	1309	12	59.90	---	---
25	188	12	59.40	---	---
24	244	13	---	40.50	---
24	244	13	---	32.07	---
24	1186	13	---	37.59	---
15	965	15	---	38.91	---
25	1869	15	---	38.40	---
1	396	17	58.00	---	---
1	396	17	59.40	---	---
1	814	17	57.40	---	---
1	814	17	58.10	---	---
1	1368	17	60.30	---	---
1	1368	17	61.40	---	---
3	527	17	59.10	---	---
18	885	17	59.41	---	---
3	525	18	---	---	0.13
3	527	18	---	---	0.14
3	535	18	---	---	0.10
3	538	18	---	---	0.13
13	56	20	59.62	---	---
13	253	20	59.05	---	---
13	375	20	59.41	---	---
13	400	20	59.33	---	---
13	624	20	59.60	---	---
13	1016	20	59.53	---	---
13	1113	20	59.40	---	---

Silicon Nitride Reference Powder

Lab #	IEA Vial #	Method Code	Si %	N %	Si (free) %
13	1281	20	59.38	---	---
13	1315	20	59.76	---	---
13	1880	20	59.56	---	---
13	9012	20	59.61	---	---
18	577	20	58.74	---	---
24	31	20	60.20	---	---
24	244	20	60.00	---	---
24	9005	20	58.10	---	---

III.2.2.

Silicon Nitride Test Powder (SNT)

Lab #	IEA Vial #	Method Code	Si %	N %	Si (free) %
14	1380	1	60.00	---	---
6	1426	2	59.10	---	---
15	381	2	60.15	---	---
21	1385	2	60.40	---	---
21	1385	2	59.90	---	---
21	1385	2	59.60	---	---
21	1385	2	60.00	---	---
16	353	4	---	---	0.021
16	361	4	---	---	0.022
16	507	4	---	---	0.028
16	507	4	---	---	0.028
16	507	4	---	---	0.029
1	824	8	---	37.40	---
1	824	8	---	37.90	---
1	1063	8	---	38.40	---
1	1063	8	---	38.30	---
5	441	8	---	39.30	---
5	785	8	---	39.10	---
5	1234	8	---	38.80	---
5	1235	8	---	38.40	---
6	383	8	---	38.50	---
6	383	8	---	39.00	---
14	1380	8	---	38.50	---
15	381	8	---	38.10	---
18	98	8	---	32.30	---
19	101	8	---	38.02	---
19	217	8	---	37.88	---
19	621	8	---	38.27	---
19	959	8	---	38.57	---
19	1061	8	---	38.59	---
19	1324	8	---	38.53	---
19	1470	8	---	39.20	---
21	1385	8	---	38.80	---
21	1385	8	---	38.90	---
21	1385	8	---	38.70	---
21	1385	8	---	38.80	---
21	1385	8	---	38.90	---
21	1385	8	---	38.90	---
24	82	8	---	39.50	---

Silicon Nitride Test Powder

Lab #	IEA Vial #	Method Code	Si %	N %	Si (free) %
24	82	8	---	38.70	---
24	82	8	---	39.30	---
24	82	8	---	38.50	---
24	82	8	---	39.70	---
24	82	8	---	39.30	---
24	82	8	---	40.50	---
24	731	8	---	39.70	---
24	731	8	---	40.70	---
24	731	8	---	39.70	---
24	731	8	---	40.10	---
24	731	8	---	38.80	---
24	731	8	---	39.60	---
24	731	8	---	41.00	---
21	1385	9	---	---	0.070
21	1385	9	---	---	0.090
5	1279	12	59.80	---	---
5	1279	12	59.70	---	---
5	1279	12	59.80	---	---
5	10870	12	59.60	---	---
5	10870	12	59.90	---	---
5	10870	12	59.70	---	---
25	629	12	59.41	---	---
25	629	12	59.20	---	---
25	629	12	59.32	---	---
25	629	12	59.18	---	---
25	629	12	59.35	---	---
25	629	12	59.43	---	---
24	1278	13	---	38.40	---
24	1278	13	---	37.90	---
5	785	15	---	39.10	---
5	785	15	---	38.80	---
5	1234	15	---	38.70	---
5	1234	15	---	38.90	---
15	381	15	---	39.04	---
25	432	15	---	38.84	---
25	432	15	---	38.97	---
25	432	15	---	38.95	---
25	432	15	---	38.93	---

Silicon Nitride Test Powder

Lab #	IEA Vial #	Method Code	Si %	N %	Si (free) %
25	432	15	---	38.79	---
25	432	15	---	39.00	---
1	1063	17	61.60	---	---
1	1063	17	62.60	---	---
18	1480	17	58.89	---	---
18	98	20	59.46	---	---
24	90	20	59.40	---	---
24	90	20	59.10	---	---

III.2.3.

Silicon Carbide Powder (SiC)

Lab #	IEA Vial #	Method Code	Si %	C (total) %
6	102	2	63.73	---
15	65	2	70.11	---
21	2	2	69.20	---
21	2	2	69.30	---
21	2	2	69.00	---
1	238	8	---	29.012
1	238	8	---	28.928
1	238	8	---	28.984
1	238	8	---	28.897
6	101	8	---	31.460
6	101	8	---	31.450
7	251	8	---	30.100
7	251	8	---	30.100
8	264	8	---	28.960
8	265	8	---	29.450
15	65	8	---	29.340
18	91	8	---	29.700
25	53	12	69.64	---
25	53	12	69.56	---
25	53	12	69.11	---
25	53	12	69.78	---
25	53	12	69.42	---
25	53	12	69.36	---
10	1025	16	---	29.970
21	1	16	---	30.100
21	1	16	---	30.100
21	1	16	---	30.040
25	55	16	---	30.070
25	55	16	---	30.070
25	55	16	---	30.010
25	55	16	---	29.970
25	55	16	---	30.010
25	55	16	---	30.050
8	262	20	65.86	---
8	263	20	68.63	---
18	91	20	68.49	29.700

III.2.4.

Silicon Powder (Si)

Lab #	IEA Vial #	Method Code	Si %
1	167	1	99.40
6	80	2	94.06
15	11	2	98.59
5	2	20	98.90
5	2	20	98.80
5	2	20	98.60
5	58	20	98.80
5	58	20	98.90
5	58	20	98.80
8	204	20	98.19
8	205	20	94.77
18	37	20	99.00

III.2.5. Yttria-Zirconia Powder (YSZ)

Lab #	IEA Vial #	Method Code	Zr %	Y %
18	54	1	67.00	4.50
18	65	1	67.10	4.33
18	OR012	1	67.20	4.54
1	62	2	65.60	3.92
1	65	2	65.00	3.88
6	40	2	68.78	3.48
15	314	2	---	4.11
17	592	2	---	4.52
22	3	2	71.40	4.60
22	75	2	71.40	4.60
2	73	6	---	4.30
22	3	12	68.44	4.23
22	3	12	68.50	4.18
22	3	12	68.50	4.19
22	75	12	68.53	4.15
22	75	12	68.54	4.15
22	75	12	68.49	4.19
24	AF017	12	---	4.52
24	OF018	12	---	4.52
24	OV018	12	---	4.52
14	559	17	---	4.20
17	592	17	---	4.41

Table III.3 Major Nonmetallic Impurities (concentration > 100 ppm by weight)

III.3.1. Silicon Nitride Reference Powder

Lab #	IEA Vial #	Method Code	C (total) %	O %	F %
24	244	3	---	---	0.128
24	646	3	---	---	0.110
24	9005	3	---	---	0.150
25	205	3	---	---	0.170
6	1640	4	---	---	0.150
6	1677	4	---	---	0.164
23	261	6	---	2.060	---
23	290	6	---	2.370	---
23	340	6	---	2.410	---
23	439	6	---	2.310	---
23	746	6	---	2.090	---
23	750	6	---	2.150	---
23	868	6	---	2.330	---
23	1001	6	---	2.050	---
23	1209	6	---	2.290	---
23	1456	6	---	2.200	---
23	1470	6	---	2.220	---
23	1788	6	---	2.110	---
1	373	8	0.170	2.568	---
1	373	8	0.170	2.548	---
1	373	8	0.170	---	---
1	1878	8	0.150	2.561	---
1	1878	8	0.160	2.449	---
1	1878	8	0.160	2.563	---
1	1878	8	---	2.491	---
3	525	8	---	2.320	---
3	525	8	---	2.320	---
3	527	8	---	2.310	---
3	527	8	---	2.310	---
3	535	8	---	2.310	---
3	535	8	---	2.310	---
3	538	8	---	2.330	---
3	538	8	---	2.330	---
5	1620	8	---	1.900	---
5	1620	8	0.120	---	---
6	106	8	0.146	2.040	---
6	106	8	0.148	2.110	---
6	106	8	0.154	---	---
6	756	8	0.160	2.180	---

Silicon Nitride Reference Powder

Lab #	IEA Vial #	Method Code	C (total) %	O %	F %
6	756	8	0.159	2.200	---
7	29	8	0.160	---	---
7	29	8	0.160	---	---
7	29	8	0.150	---	---
7	29	8	0.170	---	---
7	29	8	0.150	---	---
7	29	8	0.150	---	---
7	29	8	0.130	---	---
7	29	8	0.140	---	---
7	29	8	0.160	---	---
7	287	8	0.130	---	---
7	287	8	0.160	---	---
7	287	8	0.130	---	---
7	287	8	0.160	---	---
7	287	8	0.130	---	---
7	287	8	0.110	---	---
7	287	8	0.130	---	---
7	287	8	0.110	---	---
7	782	8	---	2.190	---
7	782	8	---	2.250	---
7	782	8	---	2.250	---
7	1033	8	---	2.320	---
7	1033	8	---	2.270	---
7	1033	8	---	2.310	---
13	56	8	---	2.110	---
13	253	8	---	2.150	---
13	375	8	---	2.130	---
13	400	8	---	2.210	---
13	624	8	---	2.230	---
13	1016	8	---	2.130	---
13	1113	8	---	2.120	---
13	1281	8	---	2.230	---
13	1315	8	---	2.130	---
13	1880	8	---	2.200	---
13	9012	8	---	2.280	---
14	103	8	0.160	---	---
14	593	8	0.160	2.000	---
15	965	8	0.150	---	---
15	1062	8	---	2.400	---
16	512	8	0.150	1.800	---
16	512	8	0.160	2.040	---
16	1664	8	0.150	1.750	---

Silicon Nitride Reference Powder

Lab #	IEA Vial #	Method Code	C (total) %	O %	F %
16	1664	8	0.170	2.010	---
17	643	8	0.150	2.230	---
17	875	8	0.190	2.210	---
18	577	8	0.130	2.130	---
18	577	8	0.130	---	---
18	885	8	0.160	2.050	---
19	1171	8	0.190	2.090	---
21	634	8	---	2.270	---
21	634	8	---	2.250	---
21	634	8	---	2.220	---
21	634	8	---	2.230	---
21	634	8	---	2.370	---
21	634	8	---	2.180	---
22	262	8	0.180	---	---
22	262	8	0.170	---	---
22	262	8	0.180	---	---
22	262	8	---	2.130	---
22	262	8	---	2.170	---
22	262	8	---	2.220	---
22	262	8	---	2.230	---
22	262	8	---	2.210	---
22	898	8	0.170	---	---
22	898	8	0.170	---	---
22	898	8	0.170	---	---
22	898	8	---	2.140	---
22	898	8	---	2.170	---
22	898	8	0.150	---	---
22	9016	8	---	2.320	---
22	9016	8	---	2.390	---
22	9016	8	---	2.420	---
22	9016	8	---	2.350	---
22	9016	8	---	2.280	---
22	9016	8	0.150	---	---
22	9016	8	0.150	---	---
22	9016	8	0.150	---	---
24	244	8	---	2.080	---
24	1704	8	---	2.100	---
24	1709	8	0.170	---	---
25	1213	8	---	2.510	---
25	1226	8	---	2.690	---
25	1231	8	---	2.600	---
25	1877	8	0.180	---	---

Silicon Nitride Reference Powder

Lab #	IEA Vial #	Method Code	C (total) %	O %	F %
5	1620	13	---	---	0.120
14	103	13	---	---	0.120
14	593	13	---	---	0.120
21	634	13	---	---	0.076
21	634	13	---	---	0.080
24	244	13	0.052	---	---
24	1041	13	0.030	---	---
24	9005	13	0.027	---	---
24	9005	13	0.032	---	---
2	1888	16	---	1.780	---
2	1888	16	---	1.980	---
2	1888	16	---	1.930	---
3	525	16	0.270	---	---
3	527	16	0.250	---	---
3	535	16	0.280	---	---
3	538	16	0.260	---	---
21	634	16	0.330	---	---
21	634	16	0.300	---	---
21	634	16	0.310	---	---

III.3.2.

Silicon Nitride Test Powder

Lab #	IEA Vial #	Method Code	C (total) %	O %	F %
24	1272	3	---	---	0.002
24	876/1272	3	---	---	0.002
24	876/1272	3	---	---	0.002
24	876/1272	3	---	---	0.002
25	1060	3	---	---	0.007
25	1060	3	---	---	0.007
25	1060	3	---	---	0.008
25	1060	3	---	---	0.007
25	1060	3	---	---	0.008
25	1060	3	---	---	0.008
6	1426	4	---	---	0.011
8	171	5	---	---	0.000
8	1328	5	---	---	0.000
8	1360	5	---	---	0.000
23	156	6	---	1.100	---
23	297	6	---	1.170	---
23	300	6	---	1.220	---
23	346	6	---	1.130	---
23	437	6	---	1.100	---
23	1400	6	---	1.110	---
23	1401	6	---	1.240	---
23	1406	6	---	1.220	---
1	824	8	---	1.550	---
1	824	8	---	1.550	---
1	824	8	0.100	---	---
1	824	8	0.100	---	---
1	1063	8	---	2.580	---
1	1063	8	---	2.590	---
1	1063	8	0.160	---	---
1	1063	8	0.160	---	---
5	441	8	---	1.190	---
5	441	8	---	1.130	---
5	785	8	0.090	---	---
5	785	8	0.100	---	---
5	1234	8	0.090	---	---
5	1234	8	0.100	---	---
5	1235	8	---	1.120	---
5	1235	8	---	1.080	---

Silicon Nitride Test Powder

Lab #	IEA Vial #	Method Code	C (total) %	O %	F %
6	383	8	0.095	1.330	---
6	383	8	0.095	1.480	---
6	383	8	0.095	---	---
7	445	8	---	1.360	---
7	445	8	---	1.360	---
7	445	8	---	1.340	---
7	1270	8	0.190	---	---
7	1270	8	0.190	---	---
7	1270	8	0.200	---	---
7	1270	8	0.200	---	---
7	1270	8	0.220	---	---
14	897	8	0.100	---	---
14	1380	8	---	1.100	---
15	381	8	0.103	1.240	---
16	321	8	0.112	---	---
16	321	8	0.133	---	---
16	321	8	0.121	---	---
16	321	8	0.116	---	---
16	321	8	0.132	---	---
16	321	8	0.135	---	---
16	321	8	---	1.251	---
16	321	8	---	1.184	---
16	321	8	---	1.107	---
16	321	8	---	1.167	---
16	321	8	---	1.253	---
16	321	8	---	1.219	---
16	1190	8	0.131	---	---
16	1190	8	0.129	---	---
16	1190	8	0.142	---	---
16	1190	8	0.137	---	---
16	1190	8	0.146	---	---
16	1190	8	0.161	---	---
16	1190	8	---	1.253	---
16	1190	8	---	1.249	---
16	1190	8	---	1.212	---
16	1190	8	---	1.193	---
16	1190	8	---	1.203	---
16	1190	8	---	1.191	---
18	98	8	0.120	1.250	---
18	1480	8	0.160	1.200	---
19	101	8	0.130	1.230	---
19	217	8	0.140	1.190	---

Silicon Nitride Test Powder

Lab #	IEA Vial #	Method Code	C (total) %	O %	F %
19	621	8	0.110	1.220	---
19	959	8	0.160	1.230	---
19	1061	8	0.120	1.230	---
19	1324	8	0.120	1.230	---
19	1470	8	0.120	1.260	---
21	1385	8	---	1.170	---
21	1385	8	---	1.180	---
21	1385	8	---	1.210	---
21	1385	8	---	1.160	---
21	1385	8	---	1.180	---
21	1385	8	---	1.170	---
22	786	8	0.110	---	---
22	786	8	0.110	---	---
22	786	8	0.110	---	---
22	786	8	---	1.280	---
22	786	8	---	1.250	---
22	1154	8	0.130	---	---
22	1154	8	0.130	---	---
22	1154	8	0.140	---	---
22	1154	8	---	1.270	---
22	1154	8	---	1.250	---
24	32	8	0.120	---	---
24	32	8	0.120	---	---
24	32	8	0.110	---	---
24	32	8	0.100	---	---
24	32	8	0.120	---	---
24	32	8	0.110	---	---
24	32	8	0.110	---	---
24	32	8	0.140	---	---
24	82	8	---	1.040	---
24	82	8	---	1.150	---
24	82	8	---	1.180	---
24	82	8	---	1.150	---
24	82	8	---	1.110	---
24	82	8	---	1.100	---
24	82	8	---	1.150	---
24	731	8	---	1.100	---
24	731	8	---	1.090	---
24	731	8	---	1.070	---
24	731	8	---	1.130	---
24	731	8	---	1.100	---
24	731	8	---	1.140	---

Silicon Nitride Test Powder

Lab #	IEA Vial #	Method Code	C (total) %	O %	F %
24	731	8	---	1.100	---
25	75	8	0.120	---	---
25	75	8	0.120	---	---
25	75	8	0.130	---	---
25	75	8	0.130	---	---
25	75	8	0.140	---	---
25	75	8	0.140	---	---
25	919	8	---	1.338	---
25	919	8	---	1.333	---
25	919	8	---	1.303	---
25	919	8	---	1.378	---
25	919	8	---	1.353	---
5	785	13	---	---	0.004
5	785	13	---	---	0.004
5	1234	13	---	---	0.004
5	1234	13	---	---	0.004
14	1108	13	---	---	0.006
14	1380	13	---	---	0.006
21	1385	13	---	---	0.005
21	1385	13	---	---	0.005
24	90	13	0.063	---	---
24	90	13	0.059	---	---
24	598	13	0.045	---	---
24	598	13	0.046	---	---
21	1385	16	0.110	---	---
21	1385	16	0.100	---	---
21	1385	16	0.120	---	---
21	1385	16	0.100	---	---
18	98	20	0.120	---	---

III.3.3.

Silicon Carbide Powder

Lab #	IEA Vial #	Method Code	C (free) %	N %	O %	F %
6	102	4	---	---	---	0.073
8	266	6	---	---	1.370	---
8	267	6	---	---	1.580	---
23	184	6	---	---	0.760	---
23	185	6	---	---	0.730	---
23	186	6	---	---	0.730	---
23	189	6	---	---	0.750	---
23	231	6	---	---	0.780	---
23	232	6	---	---	0.760	---
23	233	6	---	---	0.750	---
23	235	6	---	---	0.740	---
2	20	8	---	---	0.675	---
2	21	8	---	---	0.675	---
6	101	8	---	0.086	0.749	---
6	101	8	---	0.078	0.733	---
6	101	8	---	0.081	0.737	---
7	246	8	---	---	0.730	---
7	246	8	---	---	0.740	---
10	39	8	0.690	---	---	---
10	43	8	0.697	---	---	---
10	47	8	0.709	---	---	---
10	47	8	---	0.145	0.825	---
15	65	8	---	0.080	0.450	---
18	91	8	---	0.065	---	---
21	1	8	---	0.069	0.690	---
21	1	8	---	0.071	0.710	---
21	1	8	---	0.071	0.700	---
22	169	8	---	0.080	0.645	---
22	172	8	---	0.075	0.640	---
25	57	8	---	---	0.765	---
25	57	8	---	---	0.739	---
25	57	8	---	---	0.748	---
25	57	8	---	---	0.746	---
25	57	8	---	---	0.720	---
25	57	8	---	---	0.744	---
25	57	8	---	---	0.722	---
25	57	8	---	---	0.738	---
25	59	8	---	---	0.716	---
25	59	8	---	---	0.749	---

Silicon Carbide Powder

Lab #	IEA Vial #	Method Code	C (free) %	N %	O %	F %
25	59	8	---	---	0.728	---
25	59	8	---	---	0.733	---
25	59	8	---	---	0.755	---
21	1011	13	---	---	---	0.042
21	1011	13	---	---	---	0.040
21	1011	13	---	---	---	0.040
10	1025	16	0.270	---	---	---
21	1	16	0.570	---	---	---
21	1	16	0.580	---	---	---
21	1	16	0.580	---	---	---
21	1	16	0.600	---	---	---
22	169	20	0.660	---	---	---
22	172	20	0.630	---	---	---

III.3.4.

Silicon Powder

Lab #	IEA Vial #	Method Code	C %	N %	O %	Al %	Fe %
1	167	1	---	---	---	0.006	0.045
16	28	1	---	---	---	0.025	0.038
16	31	1	---	---	---	0.025	0.038
18	37	1	---	---	---	0.030	0.050
19	227	1	---	---	---	0.025	0.043
19	228	1	---	---	---	0.025	0.042
19	229	1	---	---	---	0.021	0.042
19	230	1	---	---	---	0.024	0.044
19	231	1	---	---	---	0.021	0.042
19	232	1	---	---	---	0.027	0.047
19	233	1	---	---	---	0.028	0.043
19	234	1	---	---	---	0.025	0.042
19	1005	1	---	---	---	0.024	0.043
5	20	2	---	---	---	0.017	0.036
5	20	2	---	---	---	0.017	0.036
5	51	2	---	---	---	0.016	0.034
5	51	2	---	---	---	0.017	0.034
6	80	2	---	---	---	0.035	0.046
7	162	2	---	---	---	0.033	0.049
7	163	2	---	---	0.570	---	---
7	163	2	---	---	0.570	---	---
7	164	2	---	---	---	0.032	0.047
15	11	2	---	---	---	0.040	0.040
17	220	2	---	---	---	0.024	0.040
20	33	2	---	---	---	0.028	0.040
20	34	2	---	---	---	0.031	0.042
22	99	2	---	---	---	0.030	0.052
22	99	2	---	---	---	0.032	0.054
22	99	2	---	---	---	0.013	0.052
22	99	2	---	---	---	0.028	0.050
22	100	2	---	---	---	0.029	0.054
22	100	2	---	---	---	0.027	0.050
22	100	2	---	---	---	0.029	0.054
22	100	2	---	---	---	0.030	0.053
8	204	5	---	---	---	0.015	---
8	205	5	---	---	---	0.015	---
8	236	6	---	---	1.820	---	---
8	235	6	---	---	1.570	---	---
23	45	6	---	---	0.630	---	---

Silicon Powder

Lab #	IEA Vial #	Method Code	C %	N %	O %	Al %	Fe %
23	46	6	---	---	0.650	---	---
23	49	6	---	---	0.650	---	---
23	50	6	---	---	0.650	---	---
23	1019	6	---	---	0.660	---	---
23	1020	6	---	---	0.560	---	---
23	1021	6	---	---	0.690	---	---
23	1025	6	---	---	0.610	---	---
1	167	7	---	---	---	0.020	0.030
1	165	8	0.080	---	---	---	---
1	165	8	0.080	---	---	---	---
2	16	8	---	---	0.510	---	---
2	66	8	---	---	0.510	---	---
3	6	8	---	0.060	0.543	---	---
3	7	8	---	0.060	0.540	---	---
3	19	8	---	0.070	0.552	---	---
3	60	8	---	0.070	0.556	---	---
5	52	8	---	---	0.490	---	---
5	52	8	---	---	0.500	---	---
5	52	8	0.057	---	---	---	---
5	52	8	0.055	---	---	---	---
5	53	8	0.053	---	---	---	---
5	53	8	0.050	---	---	---	---
5	53	8	---	---	0.550	---	---
5	53	8	---	---	0.490	---	---
6	79	8	0.080	0.010	0.480	---	---
6	79	8	0.080	0.010	0.550	---	---
6	79	8	0.080	0.010	0.470	---	---
6	79	8	---	0.020	0.490	---	---
7	160	8	0.076	---	---	---	---
7	160	8	0.078	---	---	---	---
8	206	8	0.015	---	---	---	---
8	207	8	0.028	---	---	---	---
15	11	8	0.050	0.060	0.540	---	---
16	29	8	0.084	---	---	---	---
16	29	8	0.117	---	---	---	---
16	29	8	0.095	---	---	---	---
16	29	8	0.088	---	---	---	---
16	29	8	0.127	---	---	---	---
16	29	8	0.087	---	---	---	---
16	29	8	0.096	---	---	---	---

Silicon Powder

Lab #	IEA Vial #	Method Code	C %	N %	O %	Al %	Fe %
16	29	8	0.089	---	---	---	---
16	29	8	0.091	---	---	---	---
16	74	8	---	---	0.516	---	---
16	74	8	---	---	0.534	---	---
16	74	8	---	---	0.548	---	---
16	74	8	---	---	0.529	---	---
16	74	8	---	---	0.549	---	---
18	37	8	0.210	0.010	0.490	---	---
22	99	8	0.069	---	---	---	---
22	99	8	0.071	---	---	---	---
22	99	8	0.072	---	---	---	---
22	99	8	---	---	0.520	---	---
22	99	8	---	---	0.520	---	---
22	100	8	0.069	---	---	---	---
22	100	8	0.069	---	---	---	---
22	100	8	0.071	---	---	---	---
22	100	8	---	---	0.520	---	---
22	100	8	---	---	0.510	---	---
3	6	16	0.060	---	---	---	---
3	7	16	0.063	---	---	---	---
3	19	16	0.058	---	---	---	---
3	60	16	0.062	---	---	---	---
3	6	17	---	---	---	0.043	0.044
3	7	17	---	---	---	0.042	0.050
3	19	17	---	---	---	0.042	0.063
3	60	17	---	---	---	0.044	0.043

III.3.5.

Yttria-Zirconia Powder

Lab #	IEA Vial #	Method Code	C (total) %	N %	S %	Cl %
6	403	2	---	---	0.116	---
6	403	3	---	---	---	0.018
6	630	3	---	---	0.031	0.860
24	AFO17	3	---	---	0.021	0.025
24	OFO18	3	---	---	0.017	0.028
24	OVO18	3	---	---	0.016	0.025
8	117	5	---	---	0.200	---
8	372	5	---	---	0.200	---
8	395	5	---	---	0.200	---
2	73	6	---	---	---	0.034
14	166	8	---	---	0.090	---
14	166	8	---	---	0.090	---
15	314	8	0.037	---	0.091	---
17	5924	8	0.035	---	0.100	---
22	3	8	0.033	---	---	---
22	3	8	0.031	---	---	---
22	3	8	---	0.025	---	---
22	3	8	---	0.028	---	---
22	3	8	---	0.023	---	---
22	3	8	---	0.032	---	---
22	75	8	0.036	---	---	---
22	75	8	0.039	---	---	---
22	75	8	0.038	---	---	---
22	75	8	---	0.022	---	---
22	75	8	---	0.025	---	---
22	75	8	---	0.026	---	---
22	75	8	---	0.031	---	---
14	166	14	---	---	---	0.022

Table III.4 Data Tables on Major Metallic Impurities

III.4.1. Silicon Nitride Reference Powder

Lab #	IEA Vial #	Method Code	Al %	Ca %	Fe %
1	814	1	0.0300	0.0200	0.0100
14	103	1	0.0445	0.0200	0.0155
14	593	1	0.0450	0.0205	0.0157
16	717	1	0.0380	0.0090	0.0200
16	717	1	0.0420	0.0110	0.0260
16	1667	1	0.0350	0.0070	0.0200
16	1667	1	0.0390	0.0080	0.0220
16	1747	1	0.0360	0.0060	0.0190
16	1747	1	0.0390	0.0080	0.0220
18	885	1	0.4600	0.0200	0.2700
24	244	1	---	0.0980	0.0300
24	1182	1	nd	0.0680	0.0300
24	9005	1	---	0.0500	0.0350
1	396	2	0.0300	0.0100	0.0100
1	1358	2	0.0400	0.0100	---
1	1358	2	0.0400	0.0100	---
5	1309	2	0.0380	0.0079	0.0180
6	1640	2	0.0478	0.0142	0.0491
6	1677	2	0.0360	0.0100	0.0194
7	683	2	0.0460	0.0100	0.0220
7	683	2	0.0460	0.0100	0.0230
7	1235	2	0.0440	0.0100	0.0220
7	1235	2	0.0430	0.0090	0.0210
7	1235	2	0.0450	0.0100	0.0220
7	1235	2	0.0460	0.0100	0.0220
13	56	2	0.0400	0.0200	0.0100
13	253	2	0.0200	0.0100	---
13	375	2	0.0400	0.0100	0.0100
13	400	2	0.0400	0.0100	0.0100
13	624	2	0.0400	0.0100	0.0100
13	1016	2	0.0400	0.0200	0.0100
13	1113	2	0.0200	0.0100	---
13	1281	2	0.0400	0.0400	0.0100
13	1315	2	0.0400	0.0100	---
13	1880	2	0.0300	0.0200	0.0100
13	9012	2	0.0400	0.0100	---
15	965	2	0.0415	0.0089	0.0212
19	1171	2	0.0433	0.0089	0.0203
20	1394	2	0.0015	0.0015	0.0064
21	634	2	0.0430	0.0120	0.0230
21	634	2	* 0.0450	0.0120	0.0240

Silicon Nitride Reference Powder

Lab #	IEA Vial #	Method Code	Al %	Ca %	Fe %
21	634	2	0.0440	0.0120	0.0240
21	634	2	0.0450	0.0110	0.0240
22	262	2	---	0.0100	0.0290
22	898	2	0.0530	0.0070	0.0250
22	898	2	0.0510	0.0060	0.0260
22	898	2	0.0490	0.0060	0.0290
22	898	2	0.0510	0.0060	0.0290
22	898	2	0.0380	0.0010	0.0110
22	898	2	0.0280	0.0010	0.0120
22	898	2	0.0290	0.0010	0.0110
22	898	2	0.0340	0.0010	0.0120
22	1100	2	---	0.0110	0.0170
22	9016	2	---	0.0120	0.0260
7	1681	7	0.0600	---	0.0300
23	1744	7	0.0100	---	0.0200
5	1309	12	0.0380	---	---
3	52538	17	0.0400	0.0300	0.0400
14	593	17	0.0427	0.0081	0.0222
17	643	17	0.0400	0.0070	0.0200
17	875	17	0.0400	0.0100	0.0200
25	293	17	0.0417	---	---
25	1869	17	---	0.0094	0.0233

III.4.2. Silicon Nitride Test Powder

Lab #	IEA Vial #	Method Code	Al %	Ca %	Fe %
14	1108	1	0.0012	---	0.0056
14	1380	1	0.0010	0.0032	0.0053
16	994	1	---	---	0.0038
24	598	1	---	0.0450	0.0300
24	598	1	---	0.0290	0.0350
5	924	2	0.0003	0.0005	0.0036
5	924	2	0.0003	0.0007	0.0036
5	1090	2	0.0003	0.0004	0.0035
5	1090	2	0.0003	0.0004	0.0035
6	1426	2	---	0.0073	0.0052
7	79	2	---	---	0.0028
7	79	2	---	---	0.0035
7	79	2	---	---	0.0032
15	381	2	---	0.0002	0.0039
17	304	2	0.5042	0.0123	0.0189
18	1480	2	0.2000	0.0100	0.2000
19	101	2	0.0002	0.0002	0.0043
19	217	2	0.0002	0.0003	0.0039
19	621	2	0.0002	0.0003	0.0042
19	959	2	0.0002	0.0003	0.0041
19	1061	2	0.0002	0.0002	0.0046
19	1324	2	0.0003	0.0002	0.0043
19	1470	2	0.0003	0.0003	0.0041
20	252	2	0.0001	0.0001	0.0009
21	1385	2	---	---	0.0049
21	1385	2	---	---	0.0051
21	1385	2	---	---	0.0048
21	1385	2	---	---	0.0050
21	1385	2	---	0.0016	---
21	1385	2	---	0.0015	---
21	1385	2	---	0.0015	---
21	1385	2	---	0.0015	---
22	786	2	0.0160	0.0010	0.0110
22	786	2	0.0200	0.0020	0.0100
22	786	2	0.0220	---	0.0110
22	786	2	0.0170	---	0.0100
22	1154	2	0.0210	---	0.0100
22	1154	2	0.0220	---	0.0110
22	1154	2	0.0210	---	0.0100
22	1154	2	0.0210	---	0.0110

Silicon Nitride Test Powder

Lab #	IEA Vial #	Method Code	Al %	Ca %	Fe %
8	171	5	---	0.0007	0.0026
8	1328	5	---	0.0016	0.0078
8	1360	5	---	0.0005	0.0079
2	428	6	---	---	0.0037
7	433	7	---	---	0.0040
14	1108	17	0.0015	0.0030	0.0032
14	1380	17	0.0017	0.0028	---
25	75	17	---	---	0.0042
25	75	17	---	---	0.0044
25	75	17	---	---	0.0044
25	75	17	---	---	0.0040

III.4.3.

Silicon Carbide Powder

Lab #	IEA Vial #	Method Code	Al %	Ca %	Fe %
10	46	1	---	0.0143	0.0011
16	79	1	0.0260	0.0062	0.0017
16	80	1	0.0250	0.0063	0.0016
6	102	2	0.0262	0.0099	0.0038
15	65	2	0.0333	0.0079	0.0049
17	132	2	0.0130	0.0074	---
20	88	2	0.0190	0.1200	0.0600
20	89	2	0.1000	0.1500	0.1900
21	2	2	0.0440	0.0060	0.0014
21	2	2	0.0470	0.0058	0.0014
21	2	2	0.0460	0.0061	0.0014
22	169	2	0.0640	0.0130	0.0100
22	169	2	0.0340	0.0020	0.0110
22	169	2	0.0480	0.0270	0.0040
22	169	2	0.0240	0.0190	0.0070
22	172	2	0.0550	0.0030	0.0130
22	172	2	0.0860	0.0010	0.0130
22	172	2	0.0260	0.0190	0.0040
22	172	2	0.0660	0.0200	0.0100
8	262	5	0.0050	0.0040	---
8	263	5	0.0040	0.0060	---
1	238	7	0.0100	0.0100	---
10	1025	12	0.0260	0.0020	0.0140
25	54	17	0.0451	0.0075	0.0027
25	54	17	0.0415	0.0082	0.0025
25	54	17	0.0436	0.0072	0.0028
25	54	17	0.0464	0.0088	0.0030
25	54	17	0.0432	0.0076	0.0034
25	54	17	0.0420	0.0078	0.0016
18	91	21	0.2600	---	0.1900

III.4.4.

Silicon Powder

Lab #	IEA Vial #	Method Code	C %	N %	O %	Al %	Fe %
1	167	1	---	---	---	0.006	0.045
16	28	1	---	---	---	0.025	0.038
16	31	1	---	---	---	0.025	0.038
18	37	1	---	---	---	0.030	0.050
19	227	1	---	---	---	0.025	0.043
19	228	1	---	---	---	0.025	0.042
19	229	1	---	---	---	0.021	0.042
19	230	1	---	---	---	0.024	0.044
19	231	1	---	---	---	0.021	0.042
19	232	1	---	---	---	0.027	0.047
19	233	1	---	---	---	0.028	0.043
19	234	1	---	---	---	0.025	0.042
19	1005	1	---	---	---	0.024	0.043
5	20	2	---	---	---	0.017	0.036
5	20	2	---	---	---	0.017	0.036
5	51	2	---	---	---	0.016	0.034
5	51	2	---	---	---	0.017	0.034
6	80	2	---	---	---	0.035	0.046
7	162	2	---	---	---	0.033	0.049
7	163	2	---	---	0.570	---	---
7	163	2	---	---	0.570	---	---
7	164	2	---	---	---	0.032	0.047
15	11	2	---	---	---	0.040	0.040
17	220	2	---	---	---	0.024	0.040
20	33	2	---	---	---	0.028	0.040
20	34	2	---	---	---	0.031	0.042
22	99	2	---	---	---	0.030	0.052
22	99	2	---	---	---	0.032	0.054
22	99	2	---	---	---	0.013	0.052
22	99	2	---	---	---	0.028	0.050
22	100	2	---	---	---	0.029	0.054
22	100	2	---	---	---	0.027	0.050
22	100	2	---	---	---	0.029	0.054
22	100	2	---	---	---	0.030	0.053
8	204	5	---	---	---	0.015	---
8	205	5	---	---	---	0.015	---
8	236	6	---	---	1.820	---	---
8	235	6	---	---	1.570	---	---
23	45	6	---	---	0.630	---	---

Silicon Powder

Lab #	IEA Vial #	Method Code	C %	N %	O %	Al %	Fe %
23	46	6	---	---	0.650	---	---
23	49	6	---	---	0.650	---	---
23	50	6	---	---	0.650	---	---
23	1019	6	---	---	0.660	---	---
23	1020	6	---	---	0.560	---	---
23	1021	6	---	---	0.690	---	---
23	1025	6	---	---	0.610	---	---
1	167	7	---	---	---	0.020	0.030
1	165	8	0.080	---	---	---	---
1	165	8	0.080	---	---	---	---
2	16	8	---	---	0.510	---	---
2	66	8	---	---	0.510	---	---
3	6	8	---	0.060	0.543	---	---
3	7	8	---	0.060	0.540	---	---
3	19	8	---	0.070	0.552	---	---
3	60	8	---	0.070	0.556	---	---
5	52	8	---	---	0.490	---	---
5	52	8	---	---	0.500	---	---
5	52	8	0.057	---	---	---	---
5	52	8	0.055	---	---	---	---
5	53	8	0.053	---	---	---	---
5	53	8	0.050	---	---	---	---
5	53	8	---	---	0.550	---	---
5	53	8	---	---	0.490	---	---
6	79	8	0.080	0.010	0.480	---	---
6	79	8	0.080	0.010	0.550	---	---
6	79	8	0.080	0.010	0.470	---	---
6	79	8	---	0.020	0.490	---	---
7	160	8	0.076	---	---	---	---
7	160	8	0.078	---	---	---	---
8	206	8	0.015	---	---	---	---
8	207	8	0.028	---	---	---	---
15	11	8	0.050	0.060	0.540	---	---
16	29	8	0.084	---	---	---	---
16	29	8	0.117	---	---	---	---
16	29	8	0.095	---	---	---	---
16	29	8	0.088	---	---	---	---
16	29	8	0.127	---	---	---	---
16	29	8	0.087	---	---	---	---
16	29	8	0.096	---	---	---	---

Silicon Powder

Lab #	IEA Vial #	Method Code	C %	N %	O %	Al %	Fe %
16	29	8	0.089	---	---	---	---
16	29	8	0.091	---	---	---	---
16	74	8	---	---	0.516	---	---
16	74	8	---	---	0.534	---	---
16	74	8	---	---	0.548	---	---
16	74	8	---	---	0.529	---	---
16	74	8	---	---	0.549	---	---
18	37	8	0.210	0.010	0.490	---	---
22	99	8	0.069	---	---	---	---
22	99	8	0.071	---	---	---	---
22	99	8	0.072	---	---	---	---
22	99	8	---	---	0.520	---	---
22	99	8	---	---	0.520	---	---
22	100	8	0.069	---	---	---	---
22	100	8	0.069	---	---	---	---
22	100	8	0.071	---	---	---	---
22	100	8	---	---	0.520	---	---
22	100	8	---	---	0.510	---	---
3	6	16	0.060	---	---	---	---
3	7	16	0.063	---	---	---	---
3	19	16	0.058	---	---	---	---
3	60	16	0.062	---	---	---	---
3	6	17	---	---	---	0.043	0.044
3	7	17	---	---	---	0.042	0.050
3	19	17	---	---	---	0.042	0.063
3	60	17	---	---	---	0.044	0.043

III.4.5.

Yttria-Zirconia Powder

Lab #	IEA Vial #	Method Code	Al %	Si %	Ca %	Ti %	Fe %
14	166	1	0.035	---	0.014	0.024	---
14	407	1	0.036	---	0.040	0.025	---
18	549	1	---	0.032	0.012	---	0.150
18	654	1	---	0.026	0.014	---	0.120
18	OR012	1	---	0.033	0.014	---	0.130
19	31	1	0.070	0.010	0.010	0.060	0.010
19	82	1	0.070	0.010	0.010	0.060	0.010
19	224	1	0.070	0.010	0.010	0.050	0.010
19	297	1	0.080	0.010	0.010	0.050	0.010
19	396	1	0.070	0.010	0.010	0.050	0.010
19	428	1	0.070	0.010	0.010	0.040	0.010
19	582	1	0.070	0.010	0.010	0.060	0.010
19	OC013	1	0.070	0.010	0.010	0.040	0.010
1	629	2	0.040	0.006	---	0.026	0.003
1	659	2	0.050	0.007	---	0.028	0.004
6	403	2	0.314	0.004	---	0.018	0.002
6	630	2	0.066	---	0.034	---	---
15	314	2	0.052	---	0.011	0.024	0.005
17	5924	2	0.260	0.010	0.020	---	0.020
8	117	5	0.017	---	0.002	---	0.100
8	372	5	0.003	---	0.002	---	0.060
8	395	5	0.050	---	0.007	---	0.100
2	73	6	0.046	---	0.027	0.026	---
24	AF017	12	0.042	---	0.023	0.025	0.014
24	OF018	12	0.042	---	0.023	0.026	0.015
24	OV018	12	0.042	---	0.022	0.025	0.014
14	559	17	0.048	---	---	0.025	0.003
17	5925	17	0.250	0.010	0.020	---	0.030

Table III.5 Data Tables for Minor Nonmetallic and Metallic Impurities

III.5.1. Silicon Nitride Reference Powder

Lab #	IEA Vial #	Method Code	S %	Cl %	Na %	Mg %
14	103	1	---	---	0.0021	0.0080
14	593	1	---	---	0.0024	0.0082
16	717	1	---	---	0.0040	0.0070
16	717	1	---	---	0.0070	0.0070
16	1667	1	---	---	0.0010	0.0060
16	1667	1	---	---	0.0020	0.0060
16	1747	1	---	---	0.0010	0.0060
16	1747	1	---	---	0.0020	0.0070
24	244	1	---	---	---	0.0360
24	1182	1	---	---	---	0.0240
24	9005	1	---	---	---	0.0200
1	1358	2	---	---	---	0.0060
1	1358	2	---	---	---	0.0060
5	1309	2	---	---	---	0.0063
6	1640	2	0.0040	---	---	nd
6	1677	2	0.0323	---	0.0047	0.0065
7	683	2	---	---	---	0.0080
7	683	2	---	---	---	0.0080
7	1235	2	---	---	---	0.0073
7	1235	2	---	---	---	0.0075
7	1235	2	---	---	---	0.0075
7	1235	2	---	---	---	0.0075
13	56	2	---	---	---	0.0100
13	253	2	---	---	---	0.0100
13	375	2	---	---	---	0.0100
13	400	2	---	---	---	0.0100
13	624	2	---	---	---	0.0100
13	1016	2	---	---	---	0.0100
13	1113	2	---	---	---	0.0100
13	1281	2	---	---	0.0600	0.0200
13	1315	2	---	---	---	0.0100
13	1880	2	---	---	0.0300	0.0100
13	9012	2	---	---	---	0.0100
15	965	2	---	---	0.0012	0.0071
20	1394	2	---	---	0.0002	0.0002
22	898	2	---	---	---	0.0030
22	898	2	---	---	---	0.0030
22	898	2	---	---	---	0.0030
22	898	2	---	---	---	0.0040

Silicon Nitride Reference Powder

Lab #	IEA Vial #	Method Code	S %	Cl %	Na %	Mg %
6	1640	3	---	0.0004	---	---
6	1677	3	---	0.0065	---	---
7	1681	7	---	---	0.0008	0.0080
22	898	7	---	0.0063	---	---
22	898	7	---	0.0046	---	---
23	1744	7	---	---	---	0.0100
17	875	8	0.0110	---	---	---
14	593	17	---	---	0.0031	0.0074
17	643	17	---	---	---	0.0070
17	875	17	---	---	---	0.0070
25	1869	17	---	---	---	0.0075
24	25	20	---	---	0.0100	---
24	638	20	---	0.0100	---	---

Silicon Nitride Reference Powder

Lab #	IEA Vial #	Method Code	K %	Ti %	Cr %	Mn %
14	103	1	---	0.0015	---	---
16	717	1	---	---	---	0.0005
16	717	1	---	---	---	0.0008
16	1667	1	---	---	---	0.0005
16	1667	1	---	---	---	0.0006
16	1747	1	---	---	---	0.0007
16	1747	1	---	---	---	0.0007
6	1640	2	---	nd	nd	nd
6	1677	2	---	---	---	nd
7	683	2	---	0.0030	---	---
7	683	2	---	0.0030	---	---
7	1235	2	---	0.0029	---	---
7	1235	2	---	0.0029	---	---
7	1235	2	---	0.0029	---	---
7	1235	2	---	0.0028	---	---
15	965	2	---	0.0030	0.0003	0.0007
18	885	2	---	0.0500	---	---
19	1171	2	---	---	---	0.0006
7	1681	7	---	0.0030	---	0.0020
25	188	17	---	0.0030	---	---
24	25	20	0.0100	---	---	---

Silicon Nitride Reference Powder

Lab #	IEA Vial #	Method Code	Ni %	W %	B %
23	1744	1	---	---	0.0020
24	244	1	0.0100	---	---
24	1182	1	nd	---	---
1	1358	2	---	---	0.0100
1	1358	2	---	---	0.0200
6	1677	2	nd	---	---
15	965	2	---	---	0.0004
19	1171	2	0.0003	---	---
20	1394	2	0.0005	---	---
7	1681	7	---	---	0.0010
17	643	17	---	0.0050	---

III.5.2.

Silicon Nitride Test Powder

Lab #	IEA Vial #	Method Code	S %	Cl %	Na %	Mg %
14	1108	1	---	---	0.0080	0.0010
14	1380	1	---	---	0.0080	0.0010
24	598	1	---	---	---	0.0130
24	598	1	---	---	---	0.0090
6	1426	2	---	---	0.0119	---
15	381	2	---	---	---	0.0002
17	304	2	---	---	---	0.0083
20	252	2	---	---	0.0002	0.0014
25	1060	3	---	0.0094	---	---
25	1060	3	---	0.0088	---	---
25	1060	3	---	0.0082	---	---
25	1060	3	---	0.0082	---	---
25	1060	3	---	0.0094	---	---
25	1060	3	---	0.0082	---	---
8	171	5	0.0005	---	---	0.0001
8	1360	5	0.0001	---	0.0003	0.0001
2	428	6	---	0.0080	---	---
22	786	7	---	0.0075	---	---
22	1154	7	---	0.0056	---	---
22	1154	7	---	0.0074	---	---
5	441	13	---	0.0076	---	---
5	441	13	---	0.0072	---	---
5	1235	13	---	0.0069	---	---
5	1235	13	---	0.0072	---	---
21	1385	13	---	0.0102	---	---
21	1385	13	---	0.0100	---	---
14	1108	17	---	---	---	0.0004
24	742	20	---	---	nd	---
24	742	20	---	---	nd	---

Silicon Nitride Test Powder

Lab #	IEA Vial #	Method Code	K %	Ti %	Cr %	Mn %
14	1108	1	0.0022	---	---	---
14	1380	1	0.0020	---	---	---
16	994	1	---	---	---	0.0003
15	381	2	---	---	0.0005	0.0000
17	304	2	---	0.0149	---	---
18	1480	2	---	0.0300	---	---
19	101	2	---	---	---	0.0001
19	217	2	---	---	---	0.0000
19	621	2	---	---	---	0.0001
19	959	2	---	---	---	0.0000
19	1061	2	---	---	---	0.0000
19	1324	2	---	---	---	0.0000
19	1470	2	---	---	---	0.0001
8	171	5	0.0002	---	0.0003	---
8	1328	5	0.0002	---	0.0002	---
8	1360	5	0.0002	0.0001	0.0003	---
2	428	6	---	---	0.0006	0.0000

Silicon Nitride Test Powder

Lab #	IEA Vial #	Method Code	Ni %	W %	B %
24	598	1	0.0280	---	---
24	598	1	nd	---	---
15	381	2	0.0004	---	0.0002
19	101	2	0.0006	---	---
19	217	2	0.0006	---	---
19	621	2	0.0006	---	---
19	959	2	0.0005	---	---
19	1061	2	0.0005	---	---
19	1324	2	0.0005	---	---
19	1470	2	0.0006	---	---
20	252	2	0.0005	---	---
8	171	5	0.0001	---	---
8	1328	5	0.0001	---	---
8	1360	5	0.0004	---	---
7	433	7	0.0005	---	---

III.5.3.

Silicon Carbide Powder

Lab #	IEA Vial #	Method Code	S %	Cl %	Na %	Mg %
10	46	1	---	---	---	0.0016
16	79	1	---	---	---	0.0002
16	80	1	---	---	---	0.0002
16	87	1	---	---	0.0008	---
6	102	2	0.0044	---	0.0011	---
15	65	2	---	---	0.0024	0.0003
20	88	2	---	---	0.6000	0.0170
20	89	2	---	---	0.3000	0.0110
6	102	3	---	0.0241	---	---
8	262	5	---	---	---	0.0010
21	1011	13	---	0.0055	---	---
21	1011	13	---	0.0049	---	---
21	1011	13	---	0.0050	---	---
25	55	14	---	---	---	---
25	54	17	---	---	---	0.0003
25	54	17	---	---	---	0.0003
25	54	17	---	---	---	0.0004
25	54	17	---	---	---	0.0001

Silicon Carbide Powder

Lab #	IEA Vial #	Method Code	K %	Ti %	Cr %	Mn %
16	87	1	0.0003	---	---	---
6	102	2	---	0.0047	---	---
15	65	2	---	0.0043	0.0023	0.0001
20	88	2	---	0.0010	---	---
20	89	2	---	0.0010	---	---
10	1025	12	---	0.0060	---	---
25	54	17	---	0.0035	---	---
25	54	17	---	0.0037	---	---
25	54	17	---	0.0042	---	---
25	54	17	---	0.0044	---	---
25	54	17	---	0.0038	---	---
25	54	17	---	0.0032	---	---

Silicon Carbide Powder

Lab #	IEA Vial #	Method Code	Ni %	W %	B %
15	65	2	0.0005	---	0.0005
20	88	2	0.0050	---	---
20	89	2	0.0050	---	---
21	2	2	---	---	0.0005
21	2	2	---	---	0.0006
21	2	2	---	---	0.0006

III.5.4.

Silicon Powder

Lab #	IEA Vial #	Method Code	S %	F %	Cl %	Na %
1	167	1	---	---	---	0.0010
16	28	1	---	---	---	0.0012
6	80	2	0.0005	---	---	---
15	11	2	---	---	---	0.0012
20	33	2	---	---	---	0.0008
20	34	2	---	---	---	0.0013
6	80	3	---	---	0.0210	---
6	80	4	---	0.004	---	---
5	52	13	---	0.002	0.0037	---
5	52	13	---	0.002	0.0046	---
5	53	13	---	0.002	0.0043	---
5	53	13	---	0.002	0.0033	---

Silicon Powder

Lab #	IEA Vial #	Method Code	Mg %	K %	Ti %	Cr %
1	167	1	0.0020	0.0100	---	---
16	28	1	0.0030	0.0007	---	---
16	31	1	0.0030	0.0005	---	---
19	227	1	0.0032	---	0.0007	0.0002
19	228	1	0.0027	---	0.0007	0.0001
19	229	1	0.0022	---	0.0007	0.0001
19	230	1	0.0028	---	0.0008	0.0003
19	231	1	0.0020	---	0.0007	0.0003
19	232	1	0.0027	---	0.0007	0.0001
19	233	1	0.0031	---	0.0006	0.0001
19	234	1	0.0025	---	0.0006	0.0001
19	1005	1	0.0024	---	0.0007	0.0002
6	80	2	0.0042	---	---	---
7	162	2	---	---	0.0010	---
7	164	2	---	---	0.0010	---
15	11	2	0.0037	---	0.0007	0.0004
20	33	2	0.0029	---	0.0007	---
20	34	2	0.0074	---	0.0008	---
8	204	5	0.0030	---	---	---
3	6	17	0.0050	---	---	---
3	7	17	0.0050	---	---	---
3	19	17	0.0050	---	---	---
3	60	17	0.0050	---	---	---

Silicon Powder

Lab #	IEA Vial #	Method Code	Mn %	Ni %	W %	B %
1	167	1	0.0010	0.0010	---	0.0010
16	28	1	0.0007	---	---	---
16	31	1	0.0007	---	---	---
19	227	1	0.0007	0.0005	---	---
19	228	1	0.0007	0.0003	---	---
19	229	1	0.0007	0.0003	---	---
19	230	1	0.0007	0.0002	---	---
19	231	1	0.0007	0.0003	---	---
19	232	1	0.0008	0.0005	---	---
19	233	1	0.0007	0.0003	---	---
19	234	1	0.0007	0.0002	---	---
19	1005	1	0.0008	0.0003	---	---
7	162	2	0.0010	---	---	---
7	164	2	0.0010	0.0010	---	---
15	11	2	0.0006	0.0004	---	0.0014
20	33	2	---	0.0005	---	---
20	34	2	---	0.0005	---	---

III.5.5.

Yttria-Zirconia Powder

Lab #	IEA Vial #	Method Code	Hf %	Sn %	Nd %	Ho %
18	549	1	1.840	---	---	---
18	654	1	1.790	---	---	---
18	OR012	1	1.950	---	---	---
1	629	2	1.720	---	---	---
1	659	2	1.720	---	---	---
6	403	2	0.080	0.059	---	0.025
6	630	2	---	0.014	0.028	---
17	5924	2	1.770	---	---	---
8	117	5	0.600	---	---	---
8	372	5	0.600	---	---	---
8	395	5	0.600	---	---	---
22	3	12	1.840	---	---	---
22	3	12	1.830	---	---	---
22	3	12	1.830	---	---	---
22	75	12	1.820	---	---	---
22	75	12	1.820	---	---	---
22	75	12	1.830	---	---	---
17	5925	17	1.760	---	---	---

Yttria-Zirconia Powder

Lab #	IEA Vial #	Method Code	Na %	Mg %	K %	Ga %	Ba %
14	166	1	0.0036	0.0009	---	---	---
14	407	1	0.0039	0.0011	---	---	---
19	31	1	0.0100	0.0100	---	---	---
19	82	1	0.0100	0.0100	---	---	---
19	224	1	0.0100	0.0100	---	---	---
19	297	1	0.0100	0.0100	---	---	---
19	396	1	0.0100	0.0100	---	---	---
19	428	1	0.0100	0.0100	---	---	---
19	582	1	0.0100	0.0100	---	---	---
19	OCO13	1	0.0100	0.0100	---	---	---
24	AF017	1	0.0080	0.0050	0.0050	---	---
24	OFO18	1	0.0070	0.0050	0.0050	---	---
24	OVO18	1	0.0070	0.0050	0.0050	---	---
1	629	2	---	0.0010	---	---	--
1	659	2	---	0.0010	---	---	---
6	403	2	---	0.0093	---	0.0058	0.0330
15	314	2	0.0022	0.0060	---	---	---
17	5924	2	---	0.0030	---	---	---
8	117	5	0.0100	---	---	---	---
8	372	5	0.0500	---	---	---	---
8	395	5	0.0100	---	---	---	---
2	73	6	0.0077	---	0.0006	---	---
14	559	17	0.0070	---	---	---	---
17	5925	17	---	0.0020	---	---	---

APPENDIX IV - Phase Composition

The codes identifying the quantitative analysis method are explained in Table IV.1.

Table IV.1 X-ray Diffraction Codes

CODE	PROCEDURE
1	Peak scan, qualitative
2	Peak scan, quantitative, single phase
3	Ratio of peak heights
4	Ratio of integrated intensities
	a. ZrO_2 : Method of Garvie and Nicholson ¹
5	Modified ratio equation, either peak heights or integrated intensities
	a. $\alpha, \beta\text{-Si}_3\text{N}_4$: Method of Mencik, Short and Peters ⁵
	b. $\alpha, \beta\text{-Si}_3\text{N}_4$: calibration curve
	c. $\alpha, \beta\text{-Si}_3\text{N}_4$: Method of Gazzara and Messier ²
	d. ZrO_2 : Modified Garvie-Nicholson method
	(1) Method of Toraya et al. ³
	(2) Method of Schmid ⁴
6	Least squares, matrix of intensities
	a. SiC: Peak areas, Lorentzian shape assumed, 8x4 system of coefficients equated to 8 peak areas ⁵
7	Whole pattern methods, e.g., Rietveld refinement

Table IV.2. Phase Composition Data Organized by Powders

IV.2.1. Silicon Nitride Reference Powder

Lab	IEA Vial #	Method Code	Alpha %	Beta %	Si ₂ ON %
20	637	2	95.000	5.000	---
20	1394	2	95.000	5.000	---
22	660	2	95.000	5.000	---
22	667	2	95.402	4.598	---
22	667	2	95.434	4.566	---
16	505	3	92.800	7.200	---
16	505	3	92.900	7.100	---
16	505	3	92.100	7.900	---
16	718	3	92.000	8.000	---
16	718	3	92.900	7.100	---
16	718	3	92.500	7.500	---
21	9007	3	97.000	3.000	---
24	1669	3	93.000	7.000	---
2	1482	4	94.500	5.100	0.40
4	1344	4	92.000	5.000	---
5	230	4	93.700	6.300	---
5	1309	4	93.800	6.200	---
6	422	4	95.000	5.000	---
13	56	4	94.000	6.000	---
13	253	4	94.000	6.000	---
13	375	4	93.000	7.000	0.50
13	400	4	93.000	7.000	---
13	624	4	94.000	6.000	0.50
13	1016	4	94.000	6.000	---
13	1113	4	94.000	6.000	---
13	1281	4	94.000	6.000	---
13	1315	4	94.000	6.000	---
13	1880	4	94.000	6.000	---
13	9012	4	94.000	6.000	0.50
15	9013	4	94.000	6.000	---
25	281	4	95.700	4.300	---
25	289	4	95.300	4.700	---
25	1214	4	95.400	4.600	---

Silicon Nitride Reference Powder

Lab	IEA Vial #	Method Code	Alpha %	Beta %	Si ₂ ON %
23	499	5	90.800	6.700	---
23	1189	5	92.000	6.900	---
23	107	6	95.200	4.800	---
23	291	6	94.790	5.210	---
23	1160	6	95.180	4.820	---
23	1354	6	94.960	5.040	---
9	76	7	94.200	5.800	---
9	76	7	94.300	5.700	---
9	606	7	93.900	6.100	---
9	606	7	94.100	5.900	---
9	973	7	94.000	6.000	---
9	973	7	94.200	5.800	---
9	1475	7	93.900	6.100	---
9	1475	7	94.500	5.600	---

IV.2.2. Silicon Nitride Test Powder

Lab	IEA Vial #	Method Code	Alpha %	Beta %	Si ₂ ON %
20	252	2	97.000	3.000	---
22	60	2	97.434	2.566	---
22	60	2	97.462	2.538	---
22	237	2	97.550	2.450	---
22	237	2	97.170	2.830	---
14	723	3	96.900	3.100	---
16	382	3	94.600	5.400	---
16	382	3	95.100	4.900	---
16	382	3	94.500	5.500	---
16	916	3	95.100	4.900	---
16	916	3	95.200	4.800	---
16	916	3	95.100	4.900	---
21	LSM05	3	97.000	3.000	---
24	1093	3	95.000	5.000	---
24	1321	3	95.000	5.000	---
2	80	4	97.000	3.000	---
4	169	4	95.000	2.000	---
4	675	4	95.000	2.000	---
5	821	4	96.700	3.300	---
5	1090	4	96.400	3.600	---
6	444	4	96.000	4.000	---
15	LSM09	4	96.000	4.000	---
25	919	4	97.500	2.500	---
23	1351	5	97.100	3.000	---
23	1364	5	96.800	3.200	---
23	80	6	90.510	9.490	---
23	502	6	90.200	9.800	---
23	993	6	90.340	9.660	---
23	1813	6	90.450	9.550	---

IV.2.3.

Silicon Carbide Powder

Lab	IEA Vial #	Method Code	6H %	15R %	4H %	3C %
4	74	5	77.3	16.4	4.0	2.3
4	75	5	76.5	13.8	5.5	4.2
6	98	5	85.0	9.0	6.0	---
2	14	8	53.0	35.0	11.0	1.0

IV.2.4. Silicon

Lab	IEA Vial #	Method Code	Cubic-8F %
2	9	1	100.00
18	39	1	100.00
4	72	2	100.00
4	73	2	100.00
5	180	2	100.00
6	1003	2	100.00
20	33	2	100.00
20	34	2	100.00
22	97	2	100.00
22	97	2	100.00
22	98	2	100.00
22	98	2	100.00

IV.2.5.

Yttria-Stabilized Zirconia Powder

Lab	IEA Vial #	Method Code	Cubic %	Tetra. %	Monoc. %
9	72	2	---	84.40	15.60
9	289	2	---	84.40	15.60
9	580	2	---	84.90	15.10
22	43	2	---	87.92	12.08
2	599	3	---	82.00	18.00
14	528	3	---	80.00	20.00
24	AF017	3	---	81.00	19.00
24	OF018	3	---	78.00	22.00
24	OV018	3	---	79.00	21.00
6	191	4	---	90.60	9.40
15	OE014	4	---	83.00	17.00
18	259	4	---	79.70	20.30
18	549	4	---	80.00	20.00
18	OR012	4	---	79.30	20.70
18	OR012	4	---	80.00	20.00
18	OR012	4	---	77.00	23.00
18	OR012	4	---	75.90	24.10

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